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ANALYTICAL ABSTRACTS

1.—GENERAL ANALYTICAL CHEMISTRY

General reviews of progress, reagents and methods of general application.

1233. **Measurements without calibration curves.** V. Čupr and J. B. Pelikán (Archaeolog. Inst., Czech. Academy of Science, Prague). *Z. anal. Chem.*, 1959, **168** (5), 321–325 (in German).—By the method of standard addition it is possible to avoid the preparation of a calibration curve on the assumption that a linear relationship exists between concn. and the property measured. An expression is derived for two standard additions, one twice as great as the other, by means of which accurate results can be obtained even when the relationship is not linear. The method is illustrated by reference to the nephelometric determination of phosphate in which the results obtained with the derived expression agree with those obtained gravimetrically, while the results obtained by the normal method assuming a rectilinear relationship are low.
W. T. Carter

1234. **The [statistical] comparison of the accuracy of two analytical methods.** M. J. Maurice and B. G. Wiggers (Zent. Anal. Lab., N.V. Onderzoekinginst. "Research," Arnhem, Netherlands). *Z. anal. Chem.*, 1959, **168** (5), 335–339 (in German).—A statistical treatment is given for the comparison of the accuracy of two methods of analysis when only single determinations are available for one of of the methods. The examples chosen are the gravimetric and u.v. spectrophotometric determinations of xanthate sulphur in viscose, and it is concluded that the latter is the more accurate.
W. T. Carter

1235. **Analysis for industry [unusual reagents in redox titrimetry].** A. M. G. Macdonald. *Ind. Chem.*, 1959, **35**, 351–353.—The less common reagents in redox titrimetry are reviewed. It is pointed out that very few possess a marked advantage over conventional methods, although improved techniques have been introduced for several of these older determinations. Ascorbic acid, quinol and other reducing agents, $K_4Fe(CN)_6$, ICl, chloramine T and B and alkaline permanganate are discussed. (60 references.) S. C. I. Abstr.

1236. **Production of hydrogen sulphide for analytical purposes from mixtures of sulphur and hydrocarbons.** F. Gómez Vigide and A. Lago Hermida (Inst. "Alonso Barba," Santiago de Compostela, Spain). *Inf. Quim. Anal.*, 1959, **13** (3), 61–67.—The conditions for obtaining H_2S for analytical purposes by heating a mixture of paraffin wax and sublimed sulphur have been examined. The inclusion of kaolin brings about a more uniform evolution of gas, and the optimum mixture is sulphur - paraffin wax - kaolin (2:1:1). This gives a yield of 42% of H_2S calculated on the mixture. Equally good results are obtained by using soft paraffin as the hydrocarbon. L. A. O'Neill

1237. **Improved preparation and stability of the Karl Fischer reagent.** E. Eberius and H. Bohnes (Lab. d. A.-G. f. Zinkind., W. Grillo, Duisburg, Hamborn, Germany). *Z. anal. Chem.*, 1959, **168** (5), 330–335 (in German).—The methanol - pyridine mixture used for the preparation of the Karl Fischer reagent can be rapidly dehydrated by adding a small amount of iodine and SO_2 and adding liquid bromine until the solvent turns pale brown. The necessary amounts of iodine and SO_2 are then added to produce the reagent. Mixtures of pyridine and 2-methoxy- and 2-ethoxy-ethanol can also be dehydrated in the same way. The stability of the reagent is comparable with that made from solvents dried by the conventional methods. W. T. Carter

1238. **Studies on the metal complexes of hydroxamic acids. IV. Coloured complexes of iron and vanadium with quinaldinohydroxamic acid and their analytical uses.** R. L. Dutta (Inorg. Chem. Lab., Indian Assoc. for the Cult. of Sci., Calcutta). *J. Indian Chem. Soc.*, 1959, **36** (5), 339–345.—Quinaldinohydroxamic acid (I) was synthesised and its properties were studied. A comparison of the properties of the metal complexes of **I**, and picolino-, nicotino- and *iso*nicotino-hydroxamic acids, shows a fair amount of gradation. The complexes with **I** are characterised by comparative insolubility in water but ready solubility in organic solvents. The reagent is very sensitive for both Fe and V, and both metals can be determined spectrophotometrically in the same sample. The V complex is extracted with *iso*butyl alcohol at pH 4·0, and the Fe complex at pH 7 to 9, both being read at 450 mμ. The sensitivity for the Fe complex is 0·01 μg and that for the V complex is 0·013 μg.
I. Jones

1239. **A systematic scheme of qualitative analysis for anions. III.** I. K. Taimni and Manohar Lal (Chem. Lab., Univ. of Allahabad, India). *Anal. Chim. Acta*, 1959, **21** (1), 1–3 (in English).—In a proposed scheme for qual. analysis (*Ibid.*, 1957, **17**, 367) Zn acetate and cadmium nitrate were suggested as reagents for the separation of anions into groups. It is now shown that AsO_4^{3-}, TeO_4^{2-}, TeO_3^{2-}, WO_4^{2-}, SeO_3^{2-} and PO_3^{3-} are quant. pptd. by Zn acetate and SeO_4^{2-} by cadmium nitrate.
T. R. Andrew

1240. **Physico-chemical theories of, and new developments in apparatus for, quantitative ultramicro analysis.** M. C. Sanz (Zentrallab. des Kantonsspitals, Genf). *Chimia*, 1959, **13** (6), 192–202.—A

comprehensive review of recent developments in ultra-micro analytical apparatus and techniques is presented. The construction and use of equipment, including pipettes, burettes, shakers, titration tables, photometers, centrifuges, vessels and pH electrodes, are considered in detail. H. M.

1241. **Study of the poly(vinyl alcohol) - borate - iodine complex. I. Poly(vinyl alcohol) - boric acid as an indicator for iodometric - iodimetric titrations.** A. J. Monte-Bovi and J. J. Sciarra (Pharm. Chem. Dept., Coll. of Pharmacy, St. John's Univ., Jamaica, N.Y.). *Drug Standards*, 1959, **27** (5), 136–140.—Dissolve boric acid (25 g) in glycerol (100 ml) and use 6 drops of this soln. together with 10 drops of a 10% aq. soln. of poly(vinyl alcohol) as an indicator in iodimetric titrations. The colour changes from deep red in conc. iodine soln. through a reddish blue to colourless in the absence of iodine. The end-point is sharp to one drop of 0·1 *N* $Na_2S_2O_3$. This indicator gives results comparable with those obtained with starch in the assay of iodine, $Na_2S_2O_3$, As_2O_3, Hg_2Cl_2, $FeCl_3$ and $CuSO_4$. A. R. Rogers

1242. **Analytical chemical studies in ultra-violet light. III. Action of fluorescent adsorption indicators.** J. Bognár (Tech. Univ. for Heavy Industries, Miskolc-Egyetemváros, Hungary). *Acta Chim. Acad. Sci. Hung.*, 1959, **20** (2), 193–213.—The mechanism of the action of fluorescent adsorption indicators and of the change in their fluorescence colour was examined, with special reference to the reaction between Ag^+ and SCN^-. Some indicators [primulin, Thioflavin S, Magdala red (C.I. Basic Red 6), Indigosol blue, lactoflavin, the oxidation product of trypan red, fluorescein eosin, erythrosin, dichlorofluorescein and phloxin] form a surface compound with the ppt. on the positive surface (excess of Ag^+), and others [trypaflavin, diaminoethoxyacridine lactate, Rhodamine, 6G (C.I. Basic Red 1), Aurazine G and quinine] on the negative surface (excess of SCN^-). Thus the electronic system of the indicators becomes deformed and their fluorescence colour changes or is (usually extinguished. (See also *Magyar Kém. Foly.*, 1959, **65**, 227.) A. G. Peto

1243. **Variamine blue: a versatile redox indicator.** L. Erdey (Inst. Gen. Chem., Tech. Univ., Budapest). *Chemist Analyst*, 1959, **48** (4), 106–112.—This is a review of the numerous uses of this reagent (free base) (C.I. Azoic Diazo Component 35), with many details of procedure. (47 references.)

1244. **Tri(hydroxymethyl)methylamine as a titrimetric standard.** T. H. Whitehead (Univ. Georgia, Athens, U.S.A.). *J. Chem. Educ.*, 1959, **36** (6), 297.—The properties of tri(hydroxymethyl)methylamine (**I**) are given and its use as a primary standard for the direct standardisation of acid soln. is described. Potentiometric titration curves for Na_2CO_3 and **I**, respectively, are presented; a methyl orange - Xylene cyanol FF (C.I. Acid Blue **147**) indicator gives equally good results with both titrations, but a mixed indicator of bromocresol green and Alizarin red S (C.I. Mordant Red 3) is better. O. M. Whitton

1245. **A study of the purity and uniformity of composition of ferrous ammonium sulphate hexahydrate as a solid standard reductant.** H. J. Keily, A. Eldridge and J. O. Hibbits (Gen. Engng Lab., General Electric Co., Schenectady, N.Y., U.S.A.). *Anal. Chim. Acta*, 1959, **21** (1), 6–9 (in English).—Twelve assays on each of three 1-lb bottles of a proprietary $(NH_4)_2SO_4.FeSO_4.6H_2O$ showed the material to be of high purity (99·86 ± 0·03%) and uniformity. After storage for 10 months in an occasionally used bottle the assay fell to 99·81 ± 0·01%. T. R. Andrew

1246. **Mercuric acetate as an analytical reagent in non-aqueous titrimetry.** Kiron Kumar Kundu and Mihir Nath Das (Jadavpur Univ., Calcutta, India). *Anal. Chem.*, 1959, **31** (8), 1358–1361.—The reaction of mercuric acetate (**I**) with halogen acid salts of organic bases, or with inorganic anions, to give undissociated mercuric salts and acetate (which can be titrated with $HClO_4$) is used for the determination of cationic soaps by titration in a glycolic medium, for the analysis of binary mixtures of acetates containing **I**, for the analysis of binary mixtures of mineral acids containing HCl, and for the determination of thiols. J. P. Stern

1247. **Visual titrations in non-aqueous solvents with crystal violet as an internal indicator.** Ram Chand Paul, Jaswant Singh and Sarjit Singh Sandhu (Chem. Dept., Punjab Univ., Hoshiarpur). *J. Indian Chem. Soc.*, 1959, **36** (5), 305–310.—Acid - base titrations in non-aqueous solvents, *e.g.*, $POCl_3$, SO_2Cl_2, $AsCl_3$, CCl_4 and chlorobenzene, are described. The reversible nature of the indicator with $SnCl_4$ and $TiCl_4$ as acids, and quinoline, α-picoline and dimethylaniline as bases, in $POCl_3$ and SO_2Cl_2 is shown. With $AsCl_3$, only the basic soln. are successfully used as titrants. Carbon tetrachloride and chlorobenzene do not allow the quantitative determination of acids and bases to be made in these titrations, because the reaction is very slow, and the indicator is usually irreversibly adsorbed by the precipitate formed during the titration, or is chemically attacked. A difference in the mechanism of the reaction in polar and non-polar solvents is suggested. I. Jones

1248. **Bibliography of publications dealing with the polarographic method in 1958.** J. Heyrovský (Polarographic Inst., Czech. Acad. Sci., Prague). *Coll. Czech. Chem. Commun.*, 1959, **24**, Suppl. 1, 1–69.—This is a list of 774 references to publications in 1958. An additional 90 references to publications in previous years are included.

2.—INORGANIC ANALYSIS

General, determination of elements (arranged in the order of the Periodic Table), analysis of minerals and inorganic industrial products.

1249. **Adsorption and precipitation chromatography of inorganic compounds.** E. Hayek (Univ. Innsbruck, Austria). *J. Chromatography*, 1959, **2** (4), 334–353 (in German).—The literature is reviewed and the terms used are defined. (211 references.) G. Burger

1250. **Non-aqueous determination of inorganic salts.** R. H. Cundiff and P. C. Markunas (R. J. Reynolds Tobacco Co., Winston-Salem, N.C., U.S.A.). *Anal Chim. Acta*, 1959, **21** (1), 68–73 (in English).—Provided that the specimen will undergo anion exchange with the acidifying agent, salts of all types may be analysed by acidification with an excess of a strong acid, the addition of a non-aqueous solvent, and a differentiating potentiometric

titration (*cf. Anal. Abstr.*, 1960, **7**, 868). Results are presented for 18 salts. Carbonate and hydroxide impurities do not interfere. T. R. ANDREW

1251. **Determination of the solvation numbers of extracted inorganic compounds.** E. Jackwerth and H. Specker (Inst. f. Spektrochem., Dortmund-Aplerbeck). *Z. anal. Chem.*, 1959, **168** (5), 340–343 (in German).—Two methods are given. In the first a series of soln. of the cation containing a constant excess of anions are extracted with an inert organic solvent containing varied amounts of the active solvent. The concn. of cation and active solvent are chosen so that a constant number of moles is always present. After separation, the amount of cation in the organic phase is determined. By plotting this against the initial concn. the composition of the solvated complex can be read off from the position of the maximum on the curve. In the second method a soln. of the active solvent in an inactive solvent is titrated with a soln. of the cation, and the extinction of the aq. phase is followed photometrically. The extinction increases sharply at the point corresponding to the composition of the complex. The methods are illustrated by reference to the systems $UO_2(NO_3)_2$ and $Fe(SCN)_3$ with tributyl phosphate in octane. W. T. CARTER

1252. **Spectrochemical analysis of elements in complex solutions using ion-exchange adsorption.** V. K. Prokof'ev, T. M. Moroshkina and I. V. Bogdanova. *Fiz. Sb. L'vovsk. Univ.*, 1958, [4 (9)], 112–114; *Ref. Zhur., Khim.*, 1959, (18), Abstr. No. 64,085.—A procedure is developed for the determination of <0·001% of U, Th and Ce in soln. in the presence of large amounts of Fe, Al, V, Cu and other elements. The metals to be determined are adsorbed on the cationites SBS, SBSR, MFS or PEK, by static or dynamic adsorption methods, from acid or alkaline soln. Allow the soln. (10 or 100 ml) to remain in contact with 0·5 g of adsorbent for 30 min., or pass it 3 times through the cationite at a rate of 1 ml per min. Filter off the resin, add an internal standard, then dry and ignite the resin at between 500° and 600° and introduce the residue into a cavity in a carbon electrode. Bake the sample in the cavity into a regulus with an arc discharge (3 amp.) for 1 min., then transfer the regulus to a cavity in a copper electrode and excite the spectra, using an upper copper electrode sharpened to a truncated cone, in an a.c. arc at 7 amp. The analysis is carried out in an autocollimating Hilger spectrograph E-492 with a glass optical system; the lines used are U 4090·1 and Mo 4084·38, Th 4019·1 and Zr 4027·2, and Ce 3999·25 and Mo 4000·49 Å. The presence of certain elements in significant amounts has a considerable influence on the slopes of the calibration curves. C. D. KOPKIN

1253. **High-frequency titration and estimation of ions. II. Determination of barium, lead, thallium, calcium, cerium, copper and silver.** A. K. Majumdar and B. K. Mitra (Dept. of Inorg. and Anal. Chem., Jadavpur Univ., Calcutta, India). *Anal. Chim. Acta*, 1959, **21** (1), 29–33 (in English).—The high-frequency technique (Majumdar and Mukherjee, *Anal. Chim. Acta*, 1959, **20**, 511) can be applied to the volumetric determination of Pb^{2+}, Ba^{2+} and Tl^{+} with K_2CrO_4; of Ca^{2+} and Ce^{3+} with ammonium oxalate; and of Cu^{2+} and Ag^{+} with $K_4Fe(CN)_6$. The optimum conditions are about 0·15 millimole of ion and a pH of 6 to 6·5. T. R. ANDREW

1254. **Quantitative separation of copper, cadmium and zinc by precipitation of sulphides with thioacetamide.** W. H. McCurdy, jun., W. J. A. Vanden Heuvel and A. R. Casazza (Dept. of Chem., Princeton Univ., N.J., U.S.A.). *Anal. Chem.*, 1959, **31** (8), 1413–1417.—Thioacetamide is used to precipitate Cu from 1·0 to 4·0 *M* $HClO_4$, <0·04 to 0·10 mg per 100 ml remaining in soln.; Cd is quant. pptd. in 2·0 *M* $HClO_4$ and Zn at pH 5·0 to 9·0. Copper and Cd are separated either by pptn. of Cu from HCl - NaCl soln. or by co-pptn. of Cu and Cd in 2·0 *M* $HClO_4$, followed by dissolution of the $CdSO_4$ in warm 1·0 *M* HCl - 4·0 *M* NaCl. The pptn. of Cu from 2 *M* trichloroacetic acid (pH 0·3) separates Cu from Zn. Two procedures for the analysis of ternary mixtures are indicated schematically. J. P. STERN

1255. **Mutual displacement reactions of metals. I. Reduction of bismuth and copper with metallic cadmium and lead and their polarographic determination. II. Polarographic determination of small amounts of metallic zinc in zinc white. III. Rapid polarographic determination of lead in granulated slag.** P. N. Kovalenko. *Uch. Zap. Rostovsk. Univ.*, 1958, **40**, 3–21, 35–44, 57–68; *Ref. Zhur., Khim.*, 1959, (18), Abstr. No. 64,086.—*Reduction of bismuth and copper*—The rates of reduction of Bi with metallic lead and of Bi and Cu with metallic cadmium are studied as a function of the acid concn. It is shown that the simultaneous presence of Cu and Bi catalytically accelerates their reduction with metallic cadmium from a zinc electrolyte, and that Bi may be completely pptd. from a nitric acid soln. by means of metallic lead. The influence of the concn. of K citrate on the rate of pptn. of Bi with metallic lead is studied. The possibility is also shown of completely pptg. Cu in ammoniacal soln.; the reduction is a lengthy process. A combined method is evolved for the determination of Bi and Cu, consisting in their pptn. from electrolytes by other metals and subsequent polarography. *Determination of metallic zinc*—It is shown that the displacement of Cu by metallic zinc may be used for the polarographic determination of small amounts of Cu. An indirect method is developed for the polarographic determination of small amounts of metallic zinc in zinc white in the presence of H_2SO_4; the pH of the mixed copper and zinc soln. during the reaction must be within the limits 4 to 5. *Determination of lead*—A method is developed for the polarographic determination of Pb in granulated slag after its cementation with metallic zinc. Satisfactory results have been obtained in the analysis of industrial samples. C. D. KOPKIN

1256. **Use of chloramine T in analytical chemistry. II. Determination of iron, aluminium, vanadium and titanium.** P. Spacu, A. Ovanesian and D. Gavanescu (Inst. Politehnic, Bucharest). *Bul. Inst. Politeh., Bucureşti*, 1957, **19** (3–4), 183–187.—Earlier work (*Ibid.*, 1957, **18**, No. 3) relating to the determination of Zn and Mg is discussed and the procedure is applied to the indirect determination of Fe, Al, V and Ti. The metals are pptd. by 8-hydroxyquinoline (I); the ppt. is filtered off and dissolved in HCl, and the liberated I is oxidised with an excess of 0·1 *N* chloramine T, the excess being then back-titrated with 0·1 *N* $Na_2S_2O_3$ in the presence of KI. (From French summary.) S.C.I. ABSTR.

1257. **Separation of hydrogen isotopes by gas-solid chromatography.** P. L. Gant and Kang Yang (Res. and Devel. Dept., Continental Oil Co., Ponca City, Okla., U.S.A.). *Science*, 1959, **129**, 1548–1549.—The analysis of mixtures of H_2, H^3H and $^3H_2$ on a molecular sieve column is described.
H. F. W. KIRKPATRICK

1258. **Autoradiography of tritium-labelled compounds on paper chromatograms.** A. W. Rogers (Dept. Anatomy, Univ. Birmingham, England). *Nature*, 1959, **184** (Suppl. No. 10), 721.—The low energy (18 keV) of the β-particles of tritium makes conventional autoradiography impossible, and a modified, more sensitive technique is described. Graded concentrations of tritium-labelled progesterone were applied to Whatman No. 1 filter-paper and developed with the Bush A solvent system (*Biochem. J.*, 1952, **50**, 370). The strips were then dried and passed through melted Ilford *K*2 emulsion, diluted 1:1 with H_2O; the coating temperature used was 50°. Excess of emulsion was drained off and the strips were allowed to dry. They were then left in a light-proof box containing silica gel for 1 to 4 weeks at 5° and ultimately processed with X-ray materials. The method is suitable for activities of 0·1 to 5 μC of tritiated progesterone; 0·1 μC represented about 0·15 μg of progesterone in the experiment described. S. BAAR

1259. **Determination of traces of phosphorus and silica in Perhydrol and of silica in ammonium fluoride.** W. Kemula and W. Wolfram (Dept. of Phys.-chem. Anal. Methods, Inst. of Phys. Chem., Polish Acad. Sci., Warsaw). *Chem. Anal., Warsaw*, 1958, **3** (5–6), 897–904 (in Polish).—For determining phosphates in the presence of silicates in Perhydrol, Scheel's method (*Z. anal. Chem.*, 1936, **105**, 256) proved most suitable, especially for serial work. Quantities of silicates equal to or greater than those of the phosphates do not influence the results. For the determination of silicates in the presence of phosphates a modification of the method is used to determine both compounds simultaneously, silica being then found by difference.
W. B. MIASKOWSKI

1260. **Determination of alkali metals by flame photometry.** J. Dvořák and Z. Řezáč (Res. Inst. Inorg. Chem., Ústí Nad Labem, Czechoslovakia). *Chem. Listy*, 1959, **53** (6), 588–599.—A review, with 76 references. J. ZÝKA

1261. **Determination of sodium oxide in sodium. The mercury method: its utilisation in the case of very low concentrations.** L. Champeix, R. Derras and J. Duflo (Commissariat à l'Énergie Atomique, Paris). *J. Nucl. Mater.*, 1959, **2**, 113–119.—The precision of the mercury method for the determination of sodium oxide is evaluated for micro-analytical procedures. The apparatus, procedure and calibration are described. The NaOH formed was determined by flame spectroscopy. The errors introduced by the presence of NaCl and $CaCl_2$ are discussed. The results obtained are examined with respect to their reproducibility. The method can be used for the determination of <10 p.p.m. of O in sodium with satisfactory precision.
NUCL. SCI. ABSTR.

1262. **Spectrographic determination of sodium and potassium in fireclay, dinas [silica brick], magnesite and other refractory materials.** A. I. Kirichenko. *Fiz. Sb. L'vovsk. Univ.*, 1958, [4 (9)], 479–483; *Ref. Zhur., Khim.*, 1959, (18), Abstr. No. 64,093.—*Procedure*—Ignite the sample at 900°, sieve it through a fine sieve (10,000 meshes per sq. cm), mix it with $Ba(NO_3)_2$ and powdered carbon in the ratio 1:1:1, and pack the mixture tightly in the cavity of a carbon electrode. Place 2 drops of a 20% ethanolic Bakelite soln. on the electrode and dry. Excite the spectra in an a.c. arc at 4 amp. with an arc gap of 5 mm. The slit of the glass spectrograph ISP-5 with camera F-270 was illuminated through a three-lens system for 30 sec. when analysing dinas, quartzite, clays and fireclay, for 40 sec. when analysing materials high in Al_2O_3, and for 50 sec. when analysing magnesite. The spectra were photographed on Panchrome plates of sensitivity 32 GOST units. The analysis is carried out from standard calibration curves with a control standard on the co-ordinates log ($I_{anal.}$/$I_{background}$) *vs.* log *C* when the background is allowed for, or log ($I_{anal.}$/$I_{comp.}$) *vs.* log *C* when the background is not allowed for. The analytical lines are Na 5688·2 and Ba 6675·2, and K 4044·1 and Ba 6675·2 A. Prepare the standards synthetically by adding sodium or potassium carbonate and igniting at 1000°. Standards based on fireclay are used for the analysis of clays and materials containing up to 50% of Al_2O_3; for materials with a higher concn. of Al_2O_3, standards are prepared from the corresponding base material. The average error is 8%. The spectrographic method agrees with the chemical methods of analysis within the limits prescribed by GOST 2642–53. The evaporation of the samples, the addition of carbon, and the influence of the current strength are studied. C. D. KOPKIN

1263. **Determination of potassium in dilute solution and its application to the analysis of sea water.** Masayoshi Ishibashi and Tadashi Hara (Inst. for Chem. Res., Kyoto Univ., Japan). *Bull. Inst. Chem. Res., Kyoto Univ.*, 1959, **37** (3), 167–171 (in English).—The hexanitrodiphenylamine method is described for the analysis of dil. soln. of potassium salts. The main defect of the method is incomplete pptn. of the K; this may be overcome by shaking the ice-cold soln. at 15-min. intervals during the pptn. When K is being determined in the presence of other salts, *e.g.*, in sea water, some co-pptn. of the reagent takes place, necessitating re-pptn. The error is $< \pm 1\%$. G. S. ROBERTS

1264. **Systematic analysis of potassium, rubidium and caesium and its application to sea muds.** Masayoshi Ishibashi and Tadashi Hara (Inst. for Chem. Res., Kyoto Univ., Japan). *Bull. Inst. Chem. Res., Kyoto Univ.*, 1959, **37** (3), 185–190 (in English).—Two schemes are presented for the separation and determination of K, Cs and Rb, based on previously published methods (*Anal. Abstr.*, 1960, **7**, 1263, 1266). The application to the analysis of sea mud is described. G. S. ROBERTS

1265. **Spectrographic determination of rubidium and caesium in rocks and minerals by the method of additions.** G. A. Shelnina and M. M. Kler. *Fiz. Sb. L'vovsk. Univ.*, 1958 [4 (9)], 115–120; *Ref. Zhur., Khim.*, 1959, (18), Abstr. No. 64,097.—The spectra are excited in an air - acetylene flame and photographed with a slit width of 0·2 mm. The analytical lines are Rb 7947, 7800, 4215 and 4201 A; and Cs 8943, 8521, 4593 and 4555 A; the background is used as internal standard. Calibration curves are constructed from synthetic standards. The samples are decomposed with a mixture of H_2SO_4 and HF

(0·6 g) and the resulting soln. is diluted to 30 ml. The size of the additions is established by a preliminary approximate analysis using the red lines. Sodium, K ard Ca are added to the soln. of the standards to obtain reproducible analytical results. Three spectra are photographed: samples with the addition of an equal vol. of a soln. containing 0·2% of Na, K and Ca (calculated as oxides), samples with the addition of Rb and Cs, and samples with a larger addition of these elements. The reproducibility is ≃9%. The dependence of the shape of the graph on the presence of other elements is studied.

C. D. Kopkin

1266. **New method for separating rubidium and caesium from potassium.** Masayoshi Ishibashi, Toshio Yamamoto and Tadashi Hara (Inst. for Chem. Res., Kyoto Univ., Japan). *Bull. Inst. Chem. Res., Kyoto Univ.*, 1959, **37** (3), 159–166 (in English).—A new reagent, prepared from 2 parts of *A* [20·5 g of $Bi(NO_3)_3.5H_2O$, 2·0 g of $AgNO_3$ and 100 ml of 6 *N* acetic acid] and 3 parts of *B* (9 *N* $NaNO_2$), and for which the formula $Na_3AgBi(NO_2)_6$ is proposed, is reported to be completely satisfactory for the separation of Rb and Cs from K. In the presence of this reagent even traces of Rb and Cs form a yellow ppt. from a satd. soln. of potassium salt. The quant. application of the method is described.

G. S. Roberts

1267. **Gravimetric determination of copper with 5-anilino-2-mercapto-1:3:4-thiadiazole. I.** E. Popper, L. Proinov and R. Crăciuneanu. *Rev. Chim., Bucharest*, 1959, **10** (6), 349–351.—The method is based on the pptn. of the Cu as a mercaptide, which is calcined to CuO. *Procedure*—An aliquot of the copper soln. (acetate, nitrate, sulphate) is treated dropwise with a 5% soln. of the reagent in ethanol, with agitation, until the ppt. changes from a green to a yellow colour. The mixture is heated on a water bath for 10 to 15 min. and filtered. The ppt. is washed with ethanol, then with water, and calcined. The residue is treated repeatedly with H_2O_2 and re-calcined carefully until free from organic matter. The standard deviation is 0·28%.

H. Sher

1268. **Determination of copper with triethylenetetramine using a metalfluorechromic indicator.** D. H. Wilkins and L. E. Hibbs (Gen. Electric Res. Lab., Schenectady, N.Y., U.S.A.). *Talanta*, 1959, **2** (3), 201–208.—Soln. containing 1 to 60 mg of Cu can be titrated with a standard soln. of triethylenetetramine (I), calcein W (purified calcein, free from fluorescein) being used as visual indicator under u.v. light. The end-point is characterised by a brilliant green fluorescence. If the titration is made at ≃pH 7 neither Mn nor Zn interferes. The procedure is applicable to the rapid determination of Cu in brass or bronze. Copper in an aluminium alloy can be determined by titration with **I** to a spectrophotometric end-point. (*Cf.* Flaschka and Soliman, *Z. anal. Chem.*, 1957, **159**, 30.)

W. J. Baker

1269. **Spectrophotometric determination of copper with tiron.** A. K. Majumdar and C. P. Savariar (Dept. of Inorg. and Anal. Chem., Jadavpur Univ., Calcutta, India). *Anal. Chim. Acta*, 1959, **21** (1), 53–57 (in English).—Tiron reacts with Cu^{II} in slightly acid soln. (optimum pH 6·1 to 6·9) to give a water-soluble green - yellow complex with max. absorption at 375 mμ (80 p.p.m. of Cu^{II} gives an extinction of 0·3 in a 1-cm cell). Beer's law is obeyed up to 160 p.p.m. of Cu^{II} and the colour is stable for 24 hr. The interference of 17 ions is reported.

T. R. Andrew

1270. **Determination of copper and zinc in metallurgical products by automatic derivative spectrophotometric titrations.** H. V. Malmstadt and T. P. Hadjiioannou (Dept. of Chem. and Chem. Engng, Univ. of Illinois, Urbana, U.S.A.). *Anal. Chim. Acta*, 1959, **21** (1), 41–46 (in English).—The Sargent - Malmstadt "Spectro-Electro" titrator has been applied to the iodimetric determination of Cu (by using the iodine absorption at 350 to 400 mμ to follow the titration) and the determination of Cu plus Zn by titration with EDTA (disodium salt), with 1-(2-pyridylazo)-2-naphthol as an indicator for both metals. Analyses of standard brass and bronze samples (Cu, 57 to 90%; Zn, 2 to 38%) yield results for Cu within ±0·5% of the stated values and for Zn within ±0·6% of the stated values, respectively. Interfering ions are removed by pptn. with aq. NH_3 followed by filtration.

T. R. Andrew

1271. **Spectrographic determination of copper and calcium in discard slags of the copper-smelting industry.** O. D. Frenkel' and T. S. Narbutovskii. Symposium: Reports of the First Urals Convention on Spectroscopy, 1956. Sverdlovsk, Metallurgizdat, 1958, 113–116; *Ref. Zhur., Khim.*, 1959, (16), Abstr. No. 56,924.—An oval groove of depth 1 mm and width 4 mm (along the chord) is drilled in a carbon rod (6 mm diam.), and is filled with the sample ground to 200 mesh; the rod is moved at a rate of 1·5 mm per sec. The spectra are excited in an a.c. arc at 10 amp. with a carbon upper electrode and an arc gap of 1·5 mm, and are photographed in a medium quartz spectrograph with three-step reduction and a slit width of 0·015 mm; 0·18 g of Ni is added per g of sample. Calibration curves are constructed from the lines (in Å) Cu 2824·3 and Ni 2821·2, Ca 3179·9 and Ni 3145·7. To eliminate the influence of third elements, the samples and standards are diluted (1:1) with a mixture of Na_2CO_3 and carbon powder (1:1). The mean error of the analysis, from 3 spectra, is 5% for Cu and 7% for Ca. The results agree satisfactorily with those by chemical methods.

C. D. Kopkin

1272. **Some characteristics of the spectrographic analysis of brass with the "rectified" arc method of excitation.** L. I. Kiselevskii, *Inzh. Fiz. Zhur.*, 1958, **1** (6), 101–103.—A "rectified" a.c. arc discharge is obtained by including a critical resistance in the primary circuit of the h.f. transformer of the PC39 commercial source unit. The polarity of the sample is controlled by increasing the capacity of the high-frequency circuit and is indicated by a centre-zero ammeter. In the determination of lead in lead-brasses and zinc in silicon-brasses, the influence of third elements is reduced to negligible proportions by using this type of discharge with the sample at a negative polarity. The spectra are photographed in a medium spectrograph with an upper copper electrode, an arc gap of 1·5 mm, a current of 4 amp., preburn of 20 sec. and an exposure of 30 to 60 sec. depending on the type of sample.

C. H. R. Gentry

1273. **Volumetric determination of silver using thioacetamide.** D. G. Bush, C. W. Zuehlke and A. E. Ballard (Res. Lab., Eastman Kodak Co., Rochester, N.Y., U.S.A.). *Anal. Chem.*, 1959, **31** (8), 1368–1371.—Silver (5 mg), as the thiosulphate

1257. **Separation of hydrogen isotopes by gas-solid chromatography.** P. L. Gant and Kang Yang (Res. and Devel. Dept., Continental Oil Co., Ponca City, Okla., U.S.A.). *Science*, 1959, **129**, 1548–1549.—The analysis of mixtures of H_2, H^3H and $^3H_2$ on a molecular sieve column is described.
H. F. W. KIRKPATRICK

1258. **Autoradiography of tritium-labelled compounds on paper chromatograms.** A. W. Rogers (Dept. Anatomy, Univ. Birmingham, England). *Nature*, 1959, **184** (Suppl. No. 10), 721.—The low energy (18 keV) of the β-particles of tritium makes conventional autoradiography impossible, and a modified, more sensitive technique is described. Graded concentrations of tritium-labelled progesterone were applied to Whatman No. 1 filter-paper and developed with the Bush A solvent system (*Biochem. J.*, 1952, **50**, 370). The strips were then dried and passed through melted Ilford *K*2 emulsion, diluted 1:1 with H_2O; the coating temperature used was 50°. Excess of emulsion was drained off and the strips were allowed to dry. They were then left in a light-proof box containing silica gel for 1 to 4 weeks at 5° and ultimately processed with X-ray materials. The method is suitable for activities of 0·1 to 5 μC of tritiated progesterone; 0·1 μC represented about 0·15 μg of progesterone in the experiment described. S. BAAR

1259. **Determination of traces of phosphorus and silica in Perhydrol and of silica in ammonium fluoride.** W. Kemula and W. Wolfram (Dept. of Phys.-chem. Anal. Methods, Inst. of Phys. Chem., Polish Acad. Sci., Warsaw). *Chem. Anal., Warsaw*, 1958, **3** (5–6), 897–904 (in Polish).—For determining phosphates in the presence of silicates in Perhydrol, Scheel's method (*Z. anal. Chem.*, 1936, **105**, 256) proved most suitable, especially for serial work. Quantities of silicates equal to or greater than those of the phosphates do not influence the results. For the determination of silicates in the presence of phosphates a modification of the method is used to determine both compounds simultaneously, silica being then found by difference.
W. B. MIASKOWSKI

1260. **Determination of alkali metals by flame photometry.** J. Dvořák and Z. Řezáč (Res. Inst. Inorg. Chem., Ústí Nad Labem, Czechoslovakia). *Chem. Listy*, 1959, **53** (6), 588–599.—A review, with 76 references. J. ZÝKA

1261. **Determination of sodium oxide in sodium. The mercury method: its utilisation in the case of very low concentrations.** L. Champeix, R. Derras and J. Duflo (Commissariat à l'Énergie Atomique, Paris). *J. Nucl. Mater.*, 1959, **2**, 113–119.—The precision of the mercury method for the determination of sodium oxide is evaluated for microanalytical procedures. The apparatus, procedure and calibration are described. The NaOH formed was determined by flame spectroscopy. The errors introduced by the presence of NaCl and $CaCl_2$ are discussed. The results obtained are examined with respect to their reproducibility. The method can be used for the determination of <10 p.p.m. of O in sodium with satisfactory precision.
NUCL. SCI. ABSTR.

1262. **Spectrographic determination of sodium and potassium in fireclay, dinas [silica brick], magnesite and other refractory materials.** A. I. Kirichenko. *Fiz. Sb. L'vovsk. Univ.*, 1958, [4 (9)], 479–483; *Ref. Zhur., Khim.*, 1959, (18), Abstr. No. 64,093.—*Procedure*—Ignite the sample at 900°, sieve it through a fine sieve (10,000 meshes per sq. cm), mix it with $Ba(NO_3)_2$ and powdered carbon in the ratio 1:1:1, and pack the mixture tightly in the cavity of a carbon electrode. Place 2 drops of a 20% ethanolic Bakelite soln. on the electrode and dry. Excite the spectra in an a.c. arc at 4 amp. with an arc gap of 5 mm. The slit of the glass spectrograph ISP-5 with camera F-270 was illuminated through a three-lens system for 30 sec. when analysing dinas, quartzite, clays and fireclay, for 40 sec. when analysing materials high in Al_2O_3, and for 50 sec. when analysing magnesite. The spectra were photographed on Panchrome plates of sensitivity 32 GOST units. The analysis is carried out from standard calibration curves with a control standard on the co-ordinates log ($I_{anal.}/I_{background}$) *vs.* log *C* when the background is allowed for, or log ($I_{anal.}/I_{comp.}$) *vs.* log *C* when the background is not allowed for. The analytical lines are Na 5688·2 and Ba 6675·2, and K 4044·1 and Ba 6675·2 A. Prepare the standards synthetically by adding sodium or potassium carbonate and igniting at 1000°. Standards based on fireclay are used for the analysis of clays and materials containing up to 50% of Al_2O_3; for materials with a higher concn. of Al_2O_3, standards are prepared from the corresponding base material. The average error is 8%. The spectrographic method agrees with the chemical methods of analysis within the limits prescribed by GOST 2642–53. The evaporation of the samples, the addition of carbon, and the influence of the current strength are studied. C. D. KOPKIN

1263. **Determination of potassium in dilute solution and its application to the analysis of sea water.** Masayoshi Ishibashi and Tadashi Hara (Inst. for Chem. Res., Kyoto Univ., Japan). *Bull. Inst. Chem. Res., Kyoto Univ.*, 1959, **37** (3), 167–171 (in English).—The hexanitrodiphenylamine method is described for the analysis of dil. soln. of potassium salts. The main defect of the method is incomplete pptn. of the K; this may be overcome by shaking the ice-cold soln. at 15-min. intervals during the pptn. When K is being determined in the presence of other salts, *e.g.*, in sea water, some co-pptn. of the reagent takes place, necessitating re-pptn. The error is $<\pm 1\%$. G. S. ROBERTS

1264. **Systematic analysis of potassium, rubidium and caesium and its application to sea muds.** Masayoshi Ishibashi and Tadashi Hara (Inst. for Chem. Res., Kyoto Univ., Japan). *Bull. Inst. Chem. Res., Kyoto Univ.*, 1959, **37** (3), 185–190 (in English).—Two schemes are presented for the separation and determination of K, Cs and Rb, based on previously published methods (*Anal. Abstr.*, 1960, **7**, 1263, 1266). The application to the analysis of sea mud is described. G. S. ROBERTS

1265. **Spectrographic determination of rubidium and caesium in rocks and minerals by the method of additions.** G. A. Shelnina and M. M. Kler. *Fiz. Sb. L'vovsk. Univ.*, 1958 [4 (9)], 115–120; *Ref. Zhur., Khim.*, 1959, (18), Abstr. No. 64,097.—The spectra are excited in an air - acetylene flame and photographed with a slit width of 0·2 mm. The analytical lines are Rb 7947, 7800, 4215 and 4201 A; and Cs 8943, 8521, 4593 and 4555 A; the background is used as internal standard. Calibration curves are constructed from synthetic standards. The samples are decomposed with a mixture of H_2SO_4 and HF

(0·6 g) and the resulting soln. is diluted to 30 ml. The size of the additions is established by a preliminary approximate analysis using the red lines. Sodium, K and Ca are added to the soln. of the standards to obtain reproducible analytical results. Three spectra are photographed: samples with the addition of an equal vol. of a soln. containing 0·2% of Na, K and Ca (calculated as oxides), samples with the addition of Rb and Cs, and samples with a larger addition of these elements. The reproducibility is ≃9%. The dependence of the shape of the graph on the presence of other elements is studied.

C. D. KOPKIN

1266. **New method for separating rubidium and caesium from potassium.** Masayoshi Ishibashi, Toshio Yamamoto and Tadashi Hara (Inst. for Chem. Res., Kyoto Univ., Japan). *Bull. Inst. Chem. Res., Kyoto Univ.*, 1959, **37** (3), 159–166 (in English).—A new reagent, prepared from 2 parts of *A* [20·5 g of $Bi(NO_3)_3.5H_2O$, 2·0 g of $AgNO_3$ and 100 ml of 6 *N* acetic acid] and 3 parts of *B* (9 *N* $NaNO_2$), and for which the formula $Na_2AgBi(NO_2)_6$ is proposed, is reported to be completely satisfactory for the separation of Rb and Cs from K. In the presence of this reagent even traces of Rb and Cs form a yellow ppt. from a satd. soln. of potassium salt. The quant. application of the method is described.

G. S. ROBERTS

1267. **Gravimetric determination of copper with 5-anilino-2-mercapto-1:3:4-thiadiazole. I.** E. Popper, L. Proinov and R. Crăciuneanu. *Rev. Chim., Bucharest*, 1959, **10** (6), 349–351.—The method is based on the pptn. of the Cu as a mercaptide, which is calcined to CuO. *Procedure*—An aliquot of the copper soln. (acetate, nitrate, sulphate) is treated dropwise with a 5% soln. of the reagent in ethanol, with agitation, until the ppt. changes from a green to a yellow colour. The mixture is heated on a water bath for 10 to 15 min. and filtered. The ppt. is washed with ethanol, then with water, and calcined. The residue is treated repeatedly with H_2O_2 and re-calcined carefully until free from organic matter. The standard deviation is 0·28%.

H. SHER

1268. **Determination of copper with triethylenetetramine using a metalfluorechromic indicator.** D. H. Wilkins and L. E. Hibbs (Gen. Electric Res. Lab., Schenectady, N.Y., U.S.A.). *Talanta*, 1959, **2** (3), 201–208.—Soln. containing 1 to 60 mg of Cu can be titrated with a standard soln. of triethylenetetramine (**I**), calcein W (purified calcein, free from fluorescein) being used as visual indicator under u.v. light. The end-point is characterised by a brilliant green fluorescence. If the titration is made at ≃pH 7 neither Mn nor Zn interferes. The procedure is applicable to the rapid determination of Cu in brass or bronze. Copper in an aluminium alloy can be determined by titration with **I** to a spectrophotometric end-point. (*Cf.* Flaschka and Soliman, *Z. anal. Chem.*, 1957, **159**, 30.)

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complex, is determined potentiometrically by titration with thioacetamide with a silver sulphide - calomel electrode pair in the presence of halides. The accuracy is within $\pm 0{\cdot}3\%$. No preliminary separation is necessary, and EDTA is used to prevent the pptn. of the sulphides of Cu, Cd, Zn, Co, Ni, Fe^{III} and Pb, but Fe^{2+} and Hg interfere. The procedure is of particular value for photographic emulsions and chemicals. J. P. STERN

1274. **Determination of palladium, platinum, gold, lead, bismuth, antimony, copper and iron in pure silver and the determination of antimony, bismuth, lead and iron in silver - copper alloys.** A. A. Kuranov and M. M. Sviridova. Symposium: Reports of the First Urals Convention on Spectroscopy, 1956. Sverdlovsk, Metallurgizdat, 1958, 85–89; *Ref. Zhur., Khim.*, 1959, (16), Abstr. No. 56,923.—Samples prepared in the form of rods of diam. 6 mm with elliptical ends are used. The spectra are excited in an a.c. arc discharge at 12 amp. with arc gap 2 mm, and are photographed in the large spectrograph KS-55 with an exposure of 120 sec.; the slit width is 0·03 mm when analysing silver and 0·02 mm when analysing silver - copper alloys. Calibration curves are constructed without allowing for the background. The error of the analysis is 10 to 20%. The standards are prepared by fusing the pure metals with the alloys, correcting for the original content by the method of addition. The most sensitive lines of the elements are used. C. D. KOPKIN

1275. **Spectrographic analysis of high-purity gold.** A. A. Kuranov and N. P. Ruksha. Symposium: Reports of the First Urals Convention on Spectroscopy, 1956. Sverdlovsk, Metallurgizdat, 1958, 105–108; *Ref. Zhur., Khim.*, 1959, (16), Abstr. No. 56,919.—Place the sample (0·2 g) in a cavity in a carbon electrode having a constriction near the working end to reduce heat losses, and fuse into a regulus for 2 or 3 sec. in an a.c. arc discharge at 5 amp. Begin the exposure of the spectra in a large spectrograph KS-55, with a carbon upper electrode at 5 amp.; after 15 sec. reduce the current to 3 amp., and continue the exposure for a further 15 sec.; photograph the spectrum of a fresh sample of gold on an adjacent part of the plate. For the determination use the lines Ag 3280·6, Cu 3247·5, Bi 3067·7, Pb 2833·0, Sb 2598·0 and Fe 2598·3, and a calibration curve. The reproducibility of the results is 5 to15% depending on the element. Prepare standards from spectroscopically pure gold. Dilute specially prepared samples containing a high content of the elements named with high-purity gold by fusion in graphite crucibles under a layer of wood charcoal; treat the melt with HCl (1:1), roll into a ribbon and take samples for preparing the regulus. The concn. of the elements in the standard alloys is determined chemically and by spectrographic analysis of the soln., allowing for residual contamination by the method of additions. [*Cf. Fiz. Sb. L'vovsk. Univ.*, 1958, [4 (9)], 421; *Ref. Zhur., Khim.*, 1959, (18), Abstr. No. 64,161.] C. D. KOPKIN

1276. **Determinations in inorganic analysis by extraction with the aid of coloured cations. III. Determination by extraction with the aid of basic dyestuffs. A. Determination of traces of gold in the presence of platinum by methyl violet.** L. Ducret and H. Maurel (École Nat. des Ind. Agric. et Aliment., Paris, France). *Anal. Chim. Acta*, 1959, **21** (1), 74–79 (in French).—In HCl soln., at pH $\simeq 1$, Au forms a complex chloroaurate ($AuCl_4^-$) with methyl violet (**I**) which can be extracted into trichloroethylene. To 10 ml of sample soln. at $\simeq$ pH 1 add aq. **I** soln. (0·04%) (3 ml), HCl (0·05 *N*) (3 ml) and trichloroethylene (15 ml). Shake vigorously for 3 min. and measure the extinction of the organic layer at 600 mμ. A blank determination with 0·1 *N* HCl instead of the sample soln. should give an extinction $<0{\cdot}01$ (extinction $= 0{\cdot}75$ for 20 μg of Au in a 1-cm cell). Platinum, if present, gives a ppt. and interferes. Such interference can be avoided by working at $\simeq$ pH 0, although the sensitivity of the procedure is then reduced by $\simeq 10\%$. T. R. ANDREW

1277. **Spectrophotometric determination of gold with phenyl α-pyridyl ketoxime.** B. Sen (Coates Chem. Lab., Louisiana State Univ., Baton Rouge, U.S.A.). *Anal. Chim. Acta*, 1959, **21** (1), 35–40 (in English).—Phenyl α-pyridyl ketoxime, previously reported as a reagent for Pd (*Anal. Abstr.*, 1959, **6**, 4792), also reacts with Au^{3+} to give a yellow chelate soluble in $CHCl_3$. The optimum pH for extraction is 3 to 6, and a triple extraction with $CHCl_3$ is necessary. The extinction of the $CHCl_3$ extract is measured at 450 mμ (0·4 in a 1-cm cell for 8 p.p.m. of Au in the extract). Interference by Cu, Co and Ni is masked by the addition of EDTA; Ag^+, Bi^{3+} and Fe^{3+} give ppt. which are removed by filtration; oxalate, citrate, tartrate, cyanide, Br^-, I^-, Pd^{2+}, Hg^{2+} and Fe^{2+} interfere seriously. The limits of identification and dilution are 1 μg and 1 in 10^6, respectively. T. R. ANDREW

1278. **Indirect complexometric titrations of beryllium with ethylenediaminetetra-acetic acid.** Seizo Misumi and Tomitsugu Taketatsu (Chem. Dept., Fac. of Sci., Kyusyu Univ., Hakozaki, Fukuoka). *Bull. Chem. Soc. Japan*, 1959, **32** (6), 593–596 (in English).—When a satd. soln. of hexa-amminocobaltic chloride is added to a soln. of basic beryllium carbonate in ammonium carbonate, an orange-yellow ppt. is formed which has the composition $[Co(NH_3)_6][(H_2O)_2Be_2(CO_3)_2(OH)_3].3H_2O$. The indirect determination of Be is carried out by the titration of the Co in the dissolved ppt. with EDTA soln., with murexide as indicator. Under optimum conditions, 0·70 to 3·50 mg of Be can be determined. The method can be applied to the separation of Be from Fe^{3+}, Al and Mg, and to its determination, with EDTA as a masking agent. I. JONES

1279. **Determination of magnesium oxide and silica in magnesium trisilicate and calcined magnesite.** F. Hobson and W. H. Stephenson (Standards Dept., Boots Pure Drug Co. Ltd., Beeston, Nottingham, England). *Analyst*, 1959, **84**, 520–521.—The sample is heated with $HClO_4$ at fuming point for 3 hr. and the diluted filtered soln. is adjusted to a known vol. The filter-paper with the insol. residue is ignited, and SiO_2 is determined by the procedure of the B.P. 1958. An aliquot of the filtrate, buffered with NH_4Cl - aq. NH_3 soln., is titrated with standard EDTA (disodium salt) soln. to the pure blue end-point of Solochrome black WDFA (C.I. Mordant Black 11) indicator. For calcined magnesite, fuming with $HClO_4$ is reduced to 45 min., and total Mg and Ca is determined by titration as before. Calcium is then determined in another aliquot by titration with standard EDTA soln. in the presence of diethylamine and with Solochrome dark blue B (C.I. Mordant Black 17) as indicator. The method of calculation is given. Results agree well with those of the B.P. assay procedure. A. O. JONES

1280. **Potentiometric determination of calcium and magnesium.** Z. I. Ivanova and P. N. Kovalenko. *Uch. Zap. Rostovsk. Univ.*, 1958, **40**, 93–101; *Ref. Zhur., Khim.*, 1959, (18), Abstr. No. 64,099.—A new zinc - zinc phosphate electrode is proposed and studied. This is an indicator electrode for the potentiometric titration of Ca and Mg when present together in soln. with alkali-metal phosphates. The titration is carried out by the method of double addition. The use of the new electrode allows 4·8 mg of Ca per litre to be titrated with an error up to $\pm 2{\cdot}08\%$, and 7·2 mg of Mg per litre with an error up to $\pm 2{\cdot}77\%$. The determination takes 2 hr., while the usual gravimetric determination takes more than 24 hr. C. D. Kopkin

1281. **Complexometric determination of calcium and magnesium in salt [sodium chloride].** H. Gómez Velasco and J. M. Rafols-Rovira (Aprovechamientos Salineros, S.A., Barcelona). *Inf. Quim. Anal.*, 1959, **13** (3), 68–73.—The usual EDTA titration of Ca and Mg (with murexide and Eriochrome black T as indicators) is carried out. An alternative system of indicators has been examined, and the results are compared with those obtained gravimetrically. L. A. O'Neill

1282. **Complexometric determination of calcium in technical phosphoric acid.** K. Łasiewicz, B. Byczyńska and H. Zawadzka (Inst. of Sulphuric Acid and Phosphate Fertilisers, Luboń, Poland). *Chem. Anal., Warsaw*, 1958, **3** (5–6), 1041–1042 (in Polish).—The calcium content of phosphoric acid produced by the action of H_2SO_4 on apatite may be determined by titration with EDTA (disodium salt), with murexide and Naphthol green B (C.I. Acid Green 1) as indicator, after the phosphates have been pptd. with $FeCl_3$. A sample (1 to 1·5 g) containing $\simeq 50\%$ of H_3PO_4, is diluted with 100 ml of H_2O, warmed to 70°, treated with 30 to 40 ml of $FeCl_3$ soln. (10% aq.) and brought to pH 6 with 4 *N* KOH, maintaining the temp. at 70° during the pptn. The soln. is filtered, made up to 100 ml with H_2O, and treated with a small quantity of ascorbic acid, 5 ml of 4 *N* KOH, and a trace of KCN. The prepared indicator (0·1 g of murexide, 0·1 g of naphthol green B and 100 g of NaCl) is added until the soln. becomes red or reddish grey, and it is then titrated with 0·01 *M* EDTA (disodium salt) to a blue or blue - green end-point. W. B. Miaskowski

1283. **Determination of calcium oxide in fluxed agglomerate on the stylometer.** I. I. Palatnik. *Fiz. Sb. L'vovsk. Univ.*, 1958, [4 (9)], 522–524; *Ref. Zhur., Khim.*, 1959, (18), Abstr. No. 64,103.—The sample of agglomerate is mixed with copper powder in the ratio 1 : 10 and pressed into a briquette of diam. 9 mm. The spectra are excited in a condensed spark and the intensities of the lines Ca 5598·4 and N 5535·9 A are estimated visually. Standards prepared from industrial samples are used to construct the calibration curve. The results agree well with those of chemical methods. The analysis takes 7 to 10 min. C. D. Kopkin

1284. **Quantitative radiochemical analysis by ion exchange. V. Calcium, strontium and barium.** L. Wish (Naval Radiolog. Defense Lab., San Francisco). *U.S. Atomic Energy Comm.*, Rep. USNRDL-TR-341, 1959, 13 pp.—A procedure was developed for the separation of Ca, Sr and Ba from the rare-earth elements and from each other. This is similar to the ion-exchange method of Baerg and Bartholomew in which Ce, La, Sr and Ba are sequentially eluted from a Dowex 50-X12 column at 87° with soln. of ammonium α-hydroxy*iso*butyrate. By lowering the cross-linkage of the resin to 8% and changing the concn. and pH, it was possible to accomplish the analytical separation at room temp. Column runs with ^{144}Ce, ^{45}Ca, ^{85}Sr and ^{133}Ba tracers showed that the recoveries were quantitative. No significant cross contamination could be detected by γ-ray spectrometer measurements. The determination of Ba and Sr was then included in the sequential radiochemical analytical scheme for mixed fission product and actinide elements. Radionuclides of Rh and Ru were found to be contaminants. These were eliminated by a preliminary distillation of Ru and the adsorption of Rh from 0·1 *N* HCl on to an anion-exchange column. Nucl. Sci. Abstr.

1285. **Amperometric titration of barium.** H. E. Zittel, F. J. Miller and P. F. Thomason (Anal. Chem. Div., Oak Ridge Nat. Lab., Tenn., U.S.A.). *Anal. Chem.*, 1959, **31** (8), 1351–1353.—The sensitivity of the amperometric titration of Ba^{2+} with Li_2SO_4 at pH >4 is increased by the use of tetraethylammonium bromide in aq. ethanol as medium; 5×10^{-5} to $2{\cdot}5 \times 10^{-3}$ *M* Ba^{2+} can then be determined. There is no interference from NaF, NaCl, KCl, $AlCl_3$, $CaCl_2$ or $SrCl_2$, but NO_3^- and Be interfere. The error due to Pb is additive and is corrected by the subsequent titration of Pb with $Cr_2O_7^{2-}$ in the presence of $HClO_4$. The accuracy is within $\pm 8\%$ in $2{\cdot}5 \times 10^{-4}$ *M* and $\pm 0{\cdot}8\%$ in 10^{-2} *M* soln. *Procedure*—De-aerate a mixture of $2{\cdot}5 \times 10^{-2}$ *M* $BaCl_2$ (0·5 ml), 0·1 *M* ethanolic tetraethylammonium bromide (5 ml) and H_2O (4·5 ml) with N in the titration cell. Set the potential at −2·0 V *vs.* the S.C.E. and the current sensitivity at 10 μA. Titrate with $8{\cdot}8 \times 10^{-2}$ *M* Li_2SO_4, in 0·01 to 0·03-ml amounts, and stirring with N. Take a reading 2 min. after each addition. The end-point is indicated by a break in the titration curve. J. P. Stern

1286. **Polarographic determination of lead and thallium impurities when present together in zinc salts and zinc.** F. K. Baev. *Uch. Zap. Rostovsk. Univ.*, 1958, **40**, 163–172; *Ref. Zhur., Khim.*, 1959, (18), Abstr. No. 64,166.—The reduction potentials of Tl and Pb in ammoniacal tartrate soln. are studied. It is shown that in these soln. Tl and Pb give diffusion waves at potentials that differ by 0·16 to 0·29 V. The possible separation of small amounts of Pb from Tl and large amounts of Zn by pptn. of Pb as various compounds is studied. A method is developed for the polarographic determination of Tl and Pb when present together in zinc metal and its salts, based on the pptn. of Pb together with a carrier ($SrCO_3$) with ammonium carbonate from ammoniacal soln., with subsequent polarographic determination of Tl in the filtrate and Pb in the ppt. C. D. Kopkin

1287. **Polarographic determination of antimony and bismuth in zinc electrolytes.** N. A. Lektorskaya and P. N. Kovalenko. *Uch. Zap. Rostovsk. Univ.*, 1958, **40**, 173–177; *Ref. Zhur., Khim.*, 1959, (18), Abstr. No. 64,136.—A study has been made of the determination of Sb and Bi, when present together, in a basal soln. of $Zn(NO_3)_2$ in the presence of NaF and HCl. It is shown that NaF displaces the $E_{\frac{1}{2}}$ for Sb from −0·15 to −0·43 V in a basal soln. of $Zn(NO_3)_2$ in the presence of HCl (0·3 to 2·5 *N*). Under these conditions, the $E_{\frac{1}{2}}$ for Bi remains

practically unchanged (−0·09 V). Increasing the concn. of Zn in the soln. to 20% does not influence the change of $E_{\frac{1}{2}}$ for Sb and Bi, but does influence the heights of their diffusion waves; for Sb the height of the wave increases with increase in the concn. of Zn, while for Bi it decreases.

C. D. Kopkin

1288. **Rapid method for the determination of traces of nickel and cobalt in zinc electrolytes.** L. S. Nadezhdina and P. N. Kovalenko. *Uch. Zap. Rostovsk. Univ.*, 1958, **40**, 69–86; *Ref. Zhur., Khim.*, 1959, (18), Abstr. No. 64,126.—A number of organic solvents for the quant. extraction of Ni as the dimethylglyoximate from products of the zinc industry are studied, and the optimum conditions for the extraction are established. Two variations of the method for determining Ni have been developed, depending on the amounts of Mn and Fe also present. It is shown that, in the pptn. of Co as the hexanitrito complex from zinc electrolytes, Co is completely pptd. in 20 to 30 min. if its concn. in the soln. is $\nless$0·005 g per litre. The optimum conditions for the photometric determination of Co as the dimethylglyoximate are studied; this method is used as a control when determining Co in products of the zinc industry after its pptn. and concentration. Satisfactory results have been obtained in the application of the methods described to the determination of Ni and Co in synthetic mixtures and industrial samples. C. D. Kopkin

1289. **Determination of zinc in metallic cadmium.** Z. A. Tsimmergekl and Z. A. Krasnova. *Ukr. Khim. Zhur.*, 1958, **24** (6), 646–649; *Ref. Zhur., Khim.*, 1959, (16), Abstr. No. 56,844.—It is shown that Zn may be extracted from molten cadmium with fused alkali, a fused eutectic mixture of KCl and LiCl containing NH_4Cl, or fused K acetate containing ammonium acetate. The extraction of Zn from molten cadmium with the K acetate - ammonium acetate mixture is used for the rapid separation of Zn from other elements in the polarographic determination of Zn in cadmium. *Procedure*—Place K acetate (3 g) and ammonium acetate (1 g) in a heat-resistant glass tube, fuse the mixture, evaporate off the excess of water and heat to above the m.p. of cadmium. Gradually add the sample (1 to 20 g), shake for 10 min. while keeping the mixture molten and gradually adding a further 1 g of ammonium acetate; cool the tube in an inclined position, add 2 or 3 ml of water and warm to dissolve the salts, transfer the soln. to a 10-ml flask containing 2 ml of alkali soln. (10 g of NaOH, 5 g of Na_2SO_3 and 50 g of gelatin in 100 ml), make up to vol. and polarograph. If a large amount of Cd has been extracted with the Zn (the weight of metal decreasing by >6 mg), extract the Zn with *iso*amyl alcohol as the thiocyanate complex before the polarography. A control is carried out simultaneously with cadmium freed from Zn by a triple fusion under a mixture of K acetate and ammonium acetate. The described method can be used to determine 0·0002% of Zn in a 20-g sample of cadmium. C. D. Kopkin

1290. **Radiochemical separations of cadmium.** J. R. DeVoe and W. W. Meinke (Dept. of Chem., Univ. of Michigan, Ann Arbor, U.S.A.). *Anal. Chem.*, 1959, **31** (8), 1428–1432.—Four methods of separation, allowing a max. time of 30 min. per method, have been developed. The best separation from the stand-points of contamination, elements that can be separated, and time of separation, was achieved with dithizone in $CHCl_3$. Other methods included the use of ion exchange in HCl soln., and two pptn. methods, one with Reinecke salt and one with 2-*o*-hydroxyphenylbenzoxazole.

K. A. Proctor

1291. **Separation of cadmium from small amounts of zinc in nitric acid - citric acid solution with an aluminium cathode, and the polarographic determination of zinc.** P. N. Kovalenko and L. A. Ignatova. *Uch. Zap. Rostovsk. Univ.*, 1958, **40**, 127–137; *Ref. Zhur., Khim.*, 1959, (18), Abstr. No. 64,084.—A method is developed for the electrolytic separation of Cd from Zn in nitrate - citrate buffer soln. The separation may be carried out in 0·04 to 0·08 *M* Na citrate at pH 4 to 6 and a temp. of 60° to 80°; the best conditions are pH 5 and 80°; under these conditions the Zn remains completely in soln. After the separation of the Cd, the Zn is polarographed in the same soln. after adding aq. NH_3. C. D. Kopkin

1292. **Spectrographic determination of cadmium in uranium oxide by fractional pre-distillation.** L. Pszonicki (Polish Acad. Sci., Inst. of Nucl. Res., Warsaw). Inst. of Nucl. Res., Warsaw, Rep. No. 92/VIII, 1959, 15 pp.—A method was developed for determining Cd in samples of uranium, pure for nuclear purposes. In order to separate the spectra of Cd and of U and at the same time to enrich the samples, the method of fractional pre-distillation with AgCl carrier was applied. Silver chloride was found to be active during both excitation and distillation of the sample. The detection limit was 0·01 μg per g. Nucl. Sci. Abstr.

1293. **Procedure for the spectrographic analysis of metallic samples for mercury.** E. A. Sergeev and P. A. Stepanov. *Fiz. Sb. L'vovsk. Univ.*, 1958, [4 (9)], 371–374; *Ref. Zhur., Khim.*, 1959, (18), Abstr. No. 64,108.—For increasing the sensitivity of the detection of Hg, use is made of the separate evaporation of the sample and excitation of the spectra [*Ref. Zhur., Khim.*, 1956, (21), 68,676]. A steel electrode of external diam. 11 mm, with a cavity for the sample of diam. 6 mm and depth 18 mm, is fitted with a carbon cap of length 9 mm with an opening of 1·5 to 2 mm; the end of the cap is sharpened to a cone. The sample is packed tightly in the electrode to a depth of 1·5 mm (the level of the moist sample should be a little below the cap). The spectra are excited in a d.c. or a.c. arc at 10 amp. burning between the cap and a carbon upper electrode. With an exposure of 60 sec. the sensitivity of the determination by using the line 2536·5 Å is 3×10^{-5} %. The sensitivity may be increased to 1×10^{-6} % by increasing the capacity of the electrode or by heating it in an oven.

C. D. Kopkin

1294. **Carminic acid procedure for determination of boron.** D. L. Callicoat and J. D. Wolszon (Marshall Coll., Huntington, W. Va., U.S.A.). *Anal. Chem.*, 1959, **31** (8), 1434–1437.—An investigation of the variables has provided a reliable procedure accurate to within ±0·3 μg of B in the range 0 to 40 μg. The variability is traced, in part, to differences in the carminic acid - H_2SO_4 reagent soln.; careful standardisation is therefore essential. *Procedure*—Add $Ca(OH)_2$ (0·2 g) suspended in H_2O (25 ml) to the nearly neutral sample ($\ngtr$40 μg of B in $\ngtr$250 ml) in a glass flask of low boron content. Evaporate to dryness at 90°, add 40 ml of H_2SO_4 reagent [2 litres of H_2SO_4 added to a cold soln.

of 5 ml of conc. HCl in 100 to 120 ml of H_2O (the vol. of H_2O governs the time required for complete colour development)] and then 10 ml of carminic acid reagent (1 g per litre of conc. H_2SO_4), set aside at room temp. for 90 min. (or for the selected time), and measure the extinction at 610 mμ against a reagent blank. Derive the content of B from a frequently checked calibration curve, which must be prepared afresh with each new batch of reagents. J. P. STERN

1295. **Separation of microgram quantities of boron by mixed-resin-bed ion-exchange.** D. L. Callicoat, J. D. Wolszon and J. R. Hayes (Marshall Coll., Huntington, W. Va., U.S.A.). *Anal. Chem.*, 1959, **31** (8), 1437–1439.—It was found that the resin mixture Nalcite HCR (H^+ form) and Amberlite IR-45 (OH^- form) previously used for separating milligram quantities of B (*cf.* Wolszon *et al.*, *Anal. Abstr.*, 1957, **4**, 3589) gave high results with microgram amounts owing to the presence of B in the resin. By uniform elution and correction for the B found in blanks, quant. recovery of microgram quantities of B is obtained, within the limits of the carminic acid method (Callicoat and Wolszon, *Anal. Abstr.*, 1960, **7**, 1294) for determining B. Fresh resin must be used, since partly regenerated material may retain B from the sample. The salt content of the sample affects the results if the bed becomes exhausted; the procedure is thus limited to samples the total salt content of which can be controlled, and of which a comparable sample of known B content is available, *e.g.*, in the determination of B in blood ash. Corrections must be applied in the determination of B in steel. *Procedure*—Rinse the mixed resin bed (40 ml) continuously with 1 litre of H_2O at 2·5 to 2·8 ml per min. Collect the final 250 ml. Then add the sample to the column and elute with H_2O until 250 ml of eluate has been collected. Determine the B content of the eluate and the blank by the carminic acid procedure, and obtain the true B content of the sample by subtraction. J. P. STERN

1296. **Micro-determination of boron by flame photometry of a barium borotartrate complex.** E. Bovalini, L. Pucini and A. Lo Moro (Lab. de Chim., C.A.M.E.N., Livorno, Italy). *Ann. Chim., Roma*, 1959, **49** (5–6), 1046–1050.—Micro amounts of boron are pptd. as a barium borotartrate complex (*cf.* Gautier and Pignard, *Brit. Abstr. C.*, 1951, 363; Bovalini and Piazzi, *Anal. Abstr.*, 1959, **6**, 484). The ppt. is centrifuged, and washed, and the Ba is determined by flame photometry at 873 mμ, thus giving an indirect determination of boron.
C. A. FINCH

1297. **Extension of the determination of boron as a boron - curcumin complex to materials for nuclear energy use.** G. Valentini, M. L. Conti and A. Lo Moro (Lab. de Chim., C.A.M.E.N., Livorno, Italy). *Ann. Chim., Roma.*, 1959, **49** (5–6), 1039–1045.—The method of Silverman and Trego (*Anal. Abstr.*, 1957, **4**, 1203) for the micro-determination of B in uranium and its salts is applied to graphite. The sample is ignited with CaO, and the ash is dissolved in dil. HCl. Calcium is removed by treatment with Amberlite resin IR-120, and the soln. is evaporated to dryness. The residue is treated with dil. HCl, oxalic acid soln. and curcumin soln., and the extinction of the rubrocurcumin soln. in acetone or dioxan is measured at 535 mμ. Graphs of extinction *vs.* μg of B are given for acetone and dioxan soln., the slope of the former being greater than that of the latter. The slope of the graph is slightly greater when the removal of Ca is omitted. C. A. FINCH

1298. **Routine determination of boron in sedimentary rocks.** H. Werner (Chem. Lab. Geol. Landesamtes Nordrhein, Westfalen, Germany). *Z. anal. Chem.*, 1959, **168** (4), 266–268 (in German).—A modification of the method of Roth and Beck (*cf. Anal. Abstr.*, 1954, **1**, 2256) for the determination of B in fertilisers is used. *Procedure*—Fuse the sample (350 mg) containing $\not>$50 μg of B with Na_2CO_3 (1·5 g) at 875° for 1 hr. Extract with 50% (v/v) H_2SO_4 (10 ml), transfer to the distillation apparatus, add methanol - H_2SO_4 (3 : 1) (60 ml) and distil off the B in a stream of methan l vapour at 80° into 0·1 N $Ca(OH)_2$ (10 ml). Dilute to 100 ml with methanol and evaporate a 10-ml aliquot to dryness in a quartz flask. Add 10 ml of dianthrimide soln. (0·052% in conc. H_2SO_4) and a trace of solid hydrazine sulphate, and heat for 5 hr. at 70°. Measure the extinction of the soln. with a suitable photometer (Elko II, filter J62). The method has an accuracy of $\simeq\pm 10\%$ for samples containing 0·025% of B_2O_3. W. T. CARTER

1299. **Chemical determination of boron in boron carbide - zirconium clad with Zircaloy.** J. Rynasiewicz and V. F. Consalvo (Knolls Atomic Power Lab., Schenectady, N.Y.). *U.S. Atomic Energy Comm.*, Rep. KAPL-M-JR-12, 1959, 23 pp.—The sample is dissolved in HF - HCl contained in a platinum dish which is cooled in solid CO_2; $\simeq$96 to 99% of the total B remains undissolved. This is filtered off, ashed, and fused with Na_2CO_3, and the B is determined by the mannitol titration method. The acid-sol. B is determined by a modified mannitol titration after separating the Zr and other interfering cations with $BaCO_3$. The results from the two titrations are added to give the total B in the sample. The accuracy and precision of this analysis were checked on synthetic mixtures of "standard" boron carbide plus zirconium. At the 2% boron carbide level (1·59% of B) or higher, the method was accurate to at least $\pm$1·15% (relative). The precision at the 95% confidence level was $\pm$0·27 and $\pm$0·95% (relative), based on the mean of 8 measurements and a single measurement, respectively. "Standardisation" of the boron carbide sample was performed by analysing for B, C and impurities. A mass balance of 100·22 $\pm$0·69% was obtained, and the boron value was considered accurate within the precision of the analytical method. NUCL. SCI. ABSTR.

1300. **Separation and determination of micro-amounts of boron in uranium salt solutions by means of a barium borotartrate complex.** E. Bovalini, L. Pucini and M. L. Conti (Lab. de Chim., C.A.M.E.N., Livorno, Italy). *Ann. Chim., Roma*, 1959, **49** (5–6), 1051–1058.—The method described, which can be carried out without an extraction of methyl borate, comprises the pptn. of boron from ammoniacal soln. at pH 8·8 as barium borotartrate and the subsequent flame-photometric determination of the Ba. Uranium is first complexed with hydroxy-ammonium chloride. Correction curves are given for the influence of uranium and hydroxyammonium chloride. C. A. FINCH

1301. **Rapid methods of analysis. III. Complexometric determination of aluminium and chromium.** C. Liteanu, I. Crişan and C. Calu. *Rev. Chim., Bucharest*, 1959, **10** (6), 351–353.—The method has been developed for the determination

of Al and Cr in tannery soln. *Procedure for total Al plus Cr*—An aliquot of the test soln. containing $\not> 4$ mg of Cr is treated with an excess of EDTA followed by acetic acid - acetate buffer to bring the pH to $\simeq 5{\cdot}5$. After being boiled for 5 min., when it becomes red - violet, the soln. is cooled to 40°, and the excess of EDTA is titrated with 0·02 *M* $FeCl_3$ in the presence of sulphosalicylic acid, until red. The $FeCl_3$ soln. must not be too acid, and is previously buffered with acetate - acetic acid to pH $\simeq 2{\cdot}5$. *Procedure for Al alone*—The soln., previously titrated with $FeCl_3$, is acidified with HCl to pH $\simeq 1{\cdot}0$ to decompose the complex with Al^{3+}, the effect on the complexes of Fe^{3+} and Cr^{3+} being insignificant. The soln. is then titrated at 60° with the $FeCl_3$ soln. as before. Calculations are given for the pH requirements and for the results. H. SHER

1302. **Determination of aluminium in magnesium alloys by the surface-treatment method.** N. A. Tananaev and V. N. Tikhonov. *Nauch. Dokl. Vȳssh. Shkolȳ, Metallurgiya*, 1959, (1), 259–264; *Ref. Zhur., Khim.*, 1959, (17), Abstr. No. 60,491.—To small areas (bounded by paraffin wax) on the polished surfaces of sample and standard add 2 drops of H_2SO_4 (1:3); stir periodically during 5 min. with small glass rods, adding 1 drop of water three times at intervals to dilute the soln. as it becomes more concentrated. Transfer the soln. by means of capillaries to small tubes, add the washings from the treated areas, glass rods and capillaries, and transfer the resulting soln. to 10-ml measuring cylinders or 100-ml flasks, and make up to vol. To aliquots (1 or 2 ml), add a 0·2% aq. soln. of aluminon (2 drops per ml of aliquot), make up to 3 or 4 ml with the basal soln. (30 ml of 10% ammonium acetate soln., 2·5 ml of *N* HCl and 9 ml of 0·2% aluminon soln. added to 100 ml of water), set aside for 1 or 2 min., and measure by the colour comparison method, with the basal soln. as a blank. The described method can be used to analyse fabricated parts with the accuracy necessary for process control, and is very rapid (15 to 20 min.). C. D. KOPKIN

1303. **Analysis for magnesium in high-purity aluminium.** C. A. Youngdahl and F. E. DeBoer (Argonne Nat. Lab., Lemont, Ill., U.S.A.). *Nature*, 1959, **184**, 54–55.—The de-gassed sample ($\simeq 10$ g) is heated, by induction, on a graphite plate (Mg-free) at 925° to 1000° for 10 to 15 min. in a Vycor bell-jar connected to a high-vacuum system. The metal condensed on the bell-jar is dissolved in HCl and the soln. is analysed spectrographically for Mg (1 to 50 p.p.m.), Zn, Pb, Bi and Cd. This distillation method is claimed to be an improvement on Pohl's chemical method for concentrating the impurities (*cf. Anal. Abstr.*, 1954, **1**, 2062). W. J. BAKER

1304. **Complexometric determination of iron and aluminium in the control of production of aluminium compounds.** Z. Łada and I. Pacewska-Szlemińska (Anal. Lab., Inst. of Gen. Chem., Warsaw). *Chem. Anal., Warsaw*, 1958, **3** (5–6), 955–962 (in Polish).—The method, based on the titration of a sample containing Fe and Al with EDTA (disodium salt) soln., is suitable for a simultaneous determination of these metals, even when the ratios of Fe to Al vary within wide limits (1:16 to 10:1). The most important factor is the adjustment of the pH of the sample before titration. The sample in soln. is brought to pH 2 by the addition of a few drops of salicylic acid soln. (1%) and is then titrated with EDTA soln. until a yellow colour persists; this titration gives the content of Fe. A known excess of EDTA soln. is then added, and the soln. is adjusted to pH 3, warmed to 50°, cooled, and again adjusted to pH 3; 2 ml of pyridine and a few drops of catechol violet soln. (0·1%) are added and the excess of EDTA is titrated with 1·25% $CuSO_4 \cdot 5H_2O$ soln. to a blue or blue - green end-point. This titre gives Fe plus Al. The method is sufficiently accurate for routine checks of production samples, and varied quantities of SO_4^{2-} and NH_4^+ have no influence on the results. The time required is $\simeq 30$ min. W. B. MIASKOWSKI

1305. **Fluorimetric analysis. IV. Fluorimetric determination of gallium with 8-hydroxyquinoline.** Masayoshi Ishibashi, Tsunenobu Shigematsu and Yasuharu Nishikawa (Inst. for Chem. Res., Kyoto Univ., Japan). *Bull. Inst. Chem. Res., Kyoto Univ.*, 1959, **37** (3), 191–197 (in English).—The determination of Ga as the oxinate by the fluorimetric method of Sandell (*Ind. Eng. Chem., Anal. Ed.*, 1941, **13**, 844; 1947, **19**, 64) has been studied. By extraction of the oxinate into $CHCl_3$ at pH 2·6 to 2·7 only indium was found to interfere. G. S. ROBERTS

1306. **Rapid method for the determination of gallium in the products of lead and zinc manufacture.** T. T. Mityureva and A. T. Nizhnik. *Ukr. Khim. Zhur.*, 1958, **24** (6), 650–653; *Ref. Zhur., Khim.*, 1959, (16), Abstr. No. 56,849.—The influence of SO_4^{2-} and Cl^- and the H_2SO_4 concn. on the extraction of Ga as the complex of Rhodamine B with $GaCl_3$, with benzene - diethyl ether, is studied. It is shown that, except for $ZnSO_4$ and $ZnCl_2$, sulphates and chlorides do not interfere. The presence in the soln. of 10% of $ZnSO_4$ lowers the degree of extraction of the Ga by a factor of two while, with >20% of $ZnSO_4$, Ga is not extracted at all. A saturated soln. of $ZnCl_2$ in 6 *N* HCl forms an insoluble crystalline ppt. with $GaCl_3$. Small amounts of $ZnSO_4$ and $ZnCl_2$ do not interfere. The amount of Ga extracted falls as the concn. of H_2SO_4 in the soln. increases. From these results, a method is evolved for the photometric determination of Ga in intermediates and wastes of lead and zinc manufacture. *Procedure*—Decompose the sample ($\simeq 0{\cdot}1$ g) with a mixture of H_2SO_4 and HNO_3, or by fusion with an alkali-metal acid sulphate, treat the residue with hot water and filter if necessary; neutralise the filtrate with acid or alkali, add HCl sufficient to make the final soln. 6 *N* in HCl and make up to a known vol. To 2 ml of the resulting soln. add 0·5 ml of a 10% soln. of $TiCl_3$ in 6 *N* HCl, set aside for 1 or 2 min., add 0·2 to 0·5 ml of a 0·5% soln. of Rhodamine B (C.I. Basic Violet 10) in 6 *N* HCl, mix and extract twice with 2 or 3 ml of a mixture of 2 vol. of benzene and 1 vol. of ether satd. with 6 *N* HCl. Add to the organic phase (the vol. of which is measured in a 10-ml cylinder) 1 or 2 g of finely ground ignited $CaCl_2$ (to remove moisture) and measure the extinction. The results by this method and by the polarographic method do not differ by more than 5·5%. C. D. KOPKIN

1307. **Sorption of indium by ion-exchange resins from solutions containing hydrogen halides.** E. P. Tsintsevich, I. P. Alimarin and L. I. Nikolaeva. *Vestn. Moskov. Univ.*, 1959, (2), 189–197.—A study of the adsorption of In from soln. containing HCl, HBr and HI by cationite KU-2 and anionite AN-2F has been carried out with respect to (*a*) the effect of NaCl, NaBr and KI on the absorption of In at very low concn. of acid and (*b*) the effect of ethanol. It is shown that—(*i*) the indium iodide

complex ion reacts with the resins more strongly than the bromide complex; the chloride complex is the least reactive; this agrees with the results of Irving and Rossotti (*Analyst*, 1952, **77**, 801); (*ii*) the addition of a salt of a univalent ion increases the adsorption considerably at very low acid concn.; (*iii*) the addition of ethanol (40 to 80% by vol.) increases the adsorption of In. W. Roubo

1308. **Separation of thallium from accompanying elements by co-precipitation with silver iodide.** Yu. V. Morachevskiĭ, G. V. Efremov and Syuĭ Chzhigy. *Nauch. Dokl. Vȳssh. Shkolȳ. Khim. i Khim. Tekhnol.*, 1958, (4), 706–709; *Ref. Zhur., Khim.*, 1959, (15), Abstr. No. 53,126.—The radioactive isotope of Tl is used to study the recovery of small amounts of Tl^{+} from soln. by means of co-pptn. with AgI, to free it from interfering elements before its photometric determination with methyl violet. It is shown that co-pptn. takes place with excess of I^{-} in the soln.; with an excess of Ag^{+}, co-pptn. is insignificant. In HNO_3 soln. the amount of co-pptd. Tl increases as the HNO_3 concn. decreases from 5 to 0·1 *M* and reaches a max. at pH 2 to 3. At pH 3·8 the amount of Tl recovered decreases with the length of time that the ppt. is allowed to stand in contact with the mother-liquor; at pH 4 this period does not affect the recovery of Tl. With acetate buffer soln. (pH 4·7 and 6·8) and in alkaline citrate soln. (pH 9 to 10) 0·1 to 20 µg of Tl can be almost completely recovered (95 to 99%) with 70 mg of AgI; AgI is removed from the thallium ppt. by pouring hot HNO_3 (1 : 1) (20 ml) through the filter. Satisfactory recoveries were obtained of 5 to 10 µg of Tl from soln. containing 1 mg of Au, 16·5 mg of Hg^{2+}, 9 mg of Cr^{3+}, 10·3 mg of Cu^{2+} and 17 mg of Sb^{3}[?]. With large amounts of Hg^{2+}, re-pptn. with AgI is necessary as Hg^{2+} is slightly co-pptd. with AgI. K. R. C.

1309. **Determination of lanthanum and thallium in lanthanum - thallium alloys.** E. N. Kunenkova. *Trudȳ Inst. Metallurgii*, 1958, (3), 292–294; *Ref. Zhur., Khim.*, 1959, (15), Abstr. No. 53,129.—Lanthanum and Tl were determined in alloys containing 37 to 73% of Tl and 20 to 53% of La. *Procedure*—Dissolve the alloy in HNO_3, heat the soln. with $HClO_4$ to fuming, cool, dilute with H_2O and filter off the silicic acid. Add an equal vol. of 10% thiourea soln. (containing 2% of $HClO_4$) to the filtrate, set aside for 30 min., filter off the complex of Tl, wash it with 5% thiourea soln. (slightly acid) and dissolve it in warm H_2O. Neutralise the soln. with aq. NH_3 and add a further 3 ml; precipitate the Tl by boiling with an excess of 10% K_2CrO_4 soln. Wash the ppt. with 1% K_2CrO_4 soln. and then with 50% ethanol, dry at 120° to 140° and weigh as Tl_2CrO_4. Lanthanum is pptd. by oxalic acid from the filtrate and the ppt. is calcined and weighed as La_2O_3. K. R. C.

1310. **Solvent-extraction concentration of certain rare-earth elements in yttrium.** A. R. Eberle and M. W. Lerner (New Brunswick Lab., AEC, N.J.). *U.S. Atomic Energy Comm.*, Rep. AECD-4286, 1957. Decl. 1959, 10 pp.—A procedure is given for the concentration of Gd, Dy and Sm in yttrium oxide by extraction of the thiocyanates with tributyl phosphate. Up to 80-fold concentration of these elements was obtained. Results are given for the effects of NH_4SCN and tributyl phosphate concn., as well as pH, temp. and sample size on the separation. Analyses on standard yttrium oxide samples after concentration of the rare-earth elements are in good agreement with results obtained by direct spectrographic analyses. Nucl. Sci. Abstr.

1311. **Polarographic behaviour of molybdenum heteropoly acids. I. The polarography of molybdosilicic acid.** K. Grasshoff and H. Hahn (Inst. f. Anorg. Chem., Univ. Kiel, Germany). *Z. anal. Chem.*, 1959, **168** (4), 247–263 (in German).—Silica can be determined polarographically as β-molybdosilicic acid in aq. soln. containing ethyl methyl ketone and a surface-active agent as a maximum suppressor. In a Na citrate - HCl buffer at pH 2·5, the $E_{\frac{1}{2}}$ is −350 mV *vs.* the S.C.E. The max. error of the procedure over the range 0·2 to 4·0 µg of Si per ml is $\simeq \pm 3\%$ and the lower limit of detection is $\simeq$0·05 µg of Si per ml. The mechanisms of the polarographic reduction and the decomposition of β-molybdosilicic acid in aq. soln. are discussed. W. T. Carter

1312. **Colorimetric determination of phosphorus, arsenic, silicon and germanium with molybdate. I. Colorimetric determination of germanium by the molybdogermanate method.** Teru Yuasa. *Rep. Gov. Chem. Ind. Res. Inst., Tokyo*, 1959, **54** (4), 109–115.—It was found that the concn. of acid influences the colour intensity. For complete colour development, the pH of the soln., when acidified with mineral acid (H_2SO_4, HCl, HNO_3 or $HClO_4$), should be 1·5 to 2·5, and 2·0 to 4·0 when acidified with an organic acid (acetic or formic acid). The order of adding the three components, *i.e.*, germanium, acid and molybdate, has no influence on the colour intensity with the above-mentioned acids, but when the molybdate is added last, the intensity of the colour in hydroxy-acid media (*e.g.*, tartaric or citric) is appreciably diminished. Similar results are obtained by the addition of the ammonium salts of hydroxy acids. It is necessary to adjust the concn. of ammonium molybdate to at least 1 ml of 1% soln. per 25 ml when $\not>$100 µg of Ge is to be determined. Further addition of ammonium molybdate has no effect on the colour intensity.

II. The colour reaction of molybdate with reducing agent. Teru Yuasa. *Ibid.*, 1959, **54** (4), 116–120.—The coloration of molybdate by hydrazine sulphate (**I**) is studied. In acid soln., molybdate gives a blue or yellow product; the intensity of the blue colour is greatest at pH $\simeq$2·0 and does not depend on the nature of the acid. As the concn. of acid increases, the colour of the soln. changes from blue to yellow. As the time of standing or the temp. of the soln. increases, the blue colour is intensified and readily changes to yellow as the reaction proceeds. Similar results are obtained with increasing concn. of **I**. The extinction of the blue product is proportional to the amount of molybdate only when the concn. of molybdate is >5 mg per 25 ml. The yellow product obeys Beer's law within the range 0 to 20 mg of Mo per 25 ml. (From English summary.) I. Jones

1313. **Spectrophotometric determination of tin with phenylfluorone.** R. L. Bennett and H. A. Smith (Dept. of Chem., Univ. of Tennessee, Knoxville, U.S.A.). *Anal. Chem.*, 1959, **31** (8), 1441–1442.—Modifications are described to the method of Luke (*Anal. Chem.*, 1956, **28**, 1276) so as to make possible the analysis of samples containing as little as 0·02 µg of Sn per ml. The max. precision occurs with concn. ranging from 2·4 to 13 µg of Sn per 25 ml. K. A. Proctor

1314. **Determinations in inorganic analysis by extraction with the aid of coloured cations. III. Determinations by extraction with the aid of basic dyestuffs. B. Determination of traces of tin with crystal violet.** L. Ducret and H. Maurel (École Nat. des Ind. Agric. et Aliment., Paris, France). *Anal. Chim. Acta*, 1959, **21** (1), 79–85 (in French).—In HCl soln. of pH $\simeq$ 1, $SnCl_4^{2-}$ react with crystal violet (**I**) to give a product extractable with heptan-4-one. Stannic tin is reduced by Ti^{III} in the presence of cupferron. The extraction of small amounts of **I** necessitates washing the heptan-4-one extract with acid. To 5 ml of sample soln. in 1 *N* HCl add 0·5 *N* Na_2CO_3 (5 ml), heptan-4-one (5 ml), cupferron soln. (2%) (5 drops) and **I** soln. (0·3 %) (3 ml). Mix and add $TiCl_3$ soln. (35 g of 15% soln. and 150 ml of 1 *N* HCl, diluted 1 : 4 with 1 *N* HCl) (3 ml). Shake vigorously for 5 min., and reject the lower layer. Wash the organic layer with 10 ml of 0·65 *N* HCl, and measure its extinction at 595 mμ (extinction = 1·0 for $\simeq$8 μg of Sn). Tervalent Sb, Tl^{III}, Bi^{III} and Cu^{II} interfere, while Ge^{IV}, As^{III}, Pb^{II}, Co^{II}, Zn^{II} and Cr^{III} do not in concn. $< 10^{-3}$ *M*. T. R. Andrew

1315. **Determination of small amounts of tin in copper and its alloys.** H. J. G. Challis and J. T. Jones (I.C.I. Ltd., Metals Div., Kynoch Works, Witton, Birmingham, England). *Anal. Chim. Acta*, 1959, **21** (1), 58–67 (in English).—Two rapid turbidimetric methods are proposed for the determination of Sn (0·01 to 0·4%) in copper. Aluminium, As, Cd or Ni (10%), Pb, Si or Se (5%), Bi, P or Te (1%), Cr (0·5%) and Sb (0·1%) do not interfere. For Fe contents $<$0·06%, dissolve the sample (0·5 g) in HNO_3 - HCl [5 ml of a soln. containing 32 ml of HNO_3 (sp. gr. = 1·42) and 12 ml of HCl (sp. gr. = 1·18) in 100 ml], dilute to $\simeq$30 ml, heat to boiling, add 10 ml of 5% phenylarsonic acid soln. at 80°, cool to 20° and dilute to 50 ml. Set aside for 30 min. and read the extinction of the well-shaken soln. in a Spekker absorptiometer with a tungsten-filament lamp and Ilford 601 filters, or at 420 mμ in a spectrophotometer. For Fe contents up to 2%, 4-hydroxy-3-nitrophenylarsonic acid (**I**) is recommended. Dissolve 0·5 g of sample in HNO_3 - H_2SO_4 [15 ml of a mixture of 110 ml of H_2O, 50 ml of HNO_3 (sp. gr. = 1·42) and 40 ml of H_2SO_4 (sp. gr. = 1·84)], evaporate to fumes, cool, and dissolve the residue in 20 ml of H_2O. Cool to 20°, add 10 ml of **I** soln. (2%), and dilute to 50 ml. Set aside for 30 min. and measure the extinction at 470 mμ (Ilford 602 filters). Modified procedures are given for Fe contents $>$ 2% and for Sn contents of 0·001 to 0·01%. T. R. Andrew

1316. **Spectrographic determination of impurities in tin for tinning baths.** O. A. Fomina and N. S. Smirnov. Symposium: Reports of the First Urals Convention on Spectroscopy, 1956. Sverdlovsk, Metallurgizdat, 1958, 68–69; *Ref. Zhur., Khim.*, 1959, (16), Abstr. No. 56,921.—The spectra are excited in the condensed spark discharge of a generator IG-2 with a capacity of 0·01 μF and a self-inductance of 0·55 mH, and photographed in an ISP-22 spectrograph. The sample is cast into rods, the ends of which are then ground to flat surfaces. A nickel electrode sharpened to a flat cone of diam. 1 mm is used as counter electrode. The working gap is 2 mm, the secondary gap 2·5 mm. The preliminary sparking for Pb, Cu and Bi is 5 sec. with an exposure of 1 min. The determination of As, Te, Sb, Al and Zn is carried out without sparking, superimposing two photographs of the spectra in 1 min. Calibration curves are used. The analysis of 9 samples can be carried out in a day, with differences between the results of parallel determinations of 10% for Pb and 20% for the other elements. C. D. Kopkin

1317. **Absorption spectra of the hydroxyquinolinates of some less common elements. II. Photometric determination of titanium.** I. P. Alimarin, A. P. Golovina and I. V. Puzdrenkova. *Vestn. Moskov. Univ.*, 1959, (2), 185–188.—8-Hydroxyquinoline (**I**) forms a yellow complex with Ti which can be extracted by a number of org. solvents. It has been established that $CHCl_3$ is the best solvent (extracting 98·7% of the Ti) and that the extinction of the coloured extract is constant over the pH range of 1·5 to 2·5. The colour is stable for 24 hr. *Procedure*—Transfer a suitable aliquot of the test soln. (pH 1·5 to 2·5, containing 0·005 to 0·100 mg of Ti) to a separating-funnel and add 1 ml of a 2·5% $CHCl_3$ soln. of the reagent and 3 ml of $CHCl_3$ (the ratio of the aq. phase to the org. phase should be 1 : 1). Shake for 10 min., separate the org. layer and measure its extinction against $CHCl_3$ at 395 to 400 mμ. The molar absorption coefficient is 1800. The sensitivity of the reaction is 1 μg per ml; the coloured soln. obeys Beer's law over the range 0·005 to 0·100 mg of Ti. The procedure for the determination of Ti in the presence of Ce and other rare-earth elements has also been investigated. The **I** - Ce complex is not extracted at pH $\simeq$2·2 and experiments with radioactive ^{141}Ce have shown the absence of Ce in the org. phase. Take a suitable aliquot of the test soln. (pH 2·2) containing Ti and Ce^{III}, transfer it to a 25-ml separating-funnel, add 3 ml of a 2·5% soln. of the reagent in $CHCl_3$, then 3 ml of $CHCl_3$ (the ratio of the org. phase to the aq. phase should be 1 : 1). Then proceed as for the determination of Ti as described above. W. Roubo

1318. **Field method for the determination of titanium in rocks and ores.** S. I. Smÿshlaev. *Razvedka i Okhrana Nedr*, 1959, (2), 48–49; *Ref. Zhur., Khim.*, 1959, (17), Abstr. No. 60,505.—A rapid and accurate procedure is developed, based on the decomposition of silicates with NH_4F, ignition with oxalic acid, and subsequent visual colorimetry of the peroxide complex of Ti. *Procedure*—Mix the finely ground sample (20 mg) with NH_4F (0·2 to 0·5 g) in a platinum crucible, heat (350° to 450°) till the sample is completely decomposed, add oxalic acid (0·5 g) to the residue, heat, and ignite to remove oxalate; cool, moisten the walls and bottom of the crucible with H_2SO_4 (1 : 3) (2 to 3 ml), heat till no more SO_3 is given off, then add a further 2 or 3 ml of H_2SO_4 (1 : 3) and warm to give a clear soln. Transfer the soln. to a 10-ml cylinder, and wash out the crucible with water, adding the washings to the cylinder. If a residue remains in the crucible, add 0·1 g of $K_2S_2O_7$, ignite to a red heat, add 1 or 2 ml of H_2SO_4, heat, and transfer the resulting soln. to the cylinder. Make up to vol., and to 1 ml of the resulting soln. in a 10-ml cylinder add 1 drop of H_3PO_4 (1 : 1) and 3 drops of 3% H_2O_2. Match the colour with that of a standard soln. of Ti (0·2 to 0·5 mg per ml) treated similarly. The determination takes 25 to 30 min., and the error is $\not>$10%. C. D. Kopkin

1319. **Determination of titanium and iron in titaniferous materials by cerate titrimetry.** J. O. Page and A. B. Gainer (Agric. and Mech. Coll. of

Texas, College Station, U.S.A.). *Anal. Chem.*, 1959, **31** (8), 1399–1402.—After reduction of the fused sample with aluminium foil or zinc amalgam, Ti and Fe are determined successively in acid soln. by titration with $Ce(SO_4)_2$ in the absence of air, with methylene blue and N-phenylanthranilic acid, respectively, as indicators. Titanium (0·2 g) and Fe (0·09 g) are thus determined with an accuracy within $\pm$0·5 and 1·0%, respectively. Standard deviations on ilmenite ore samples are <0·31% and 0·088%, respectively. Chromium and V interfere.
J. P. STERN

1320. **Determination of oxygen and nitrogen in titanium samples by means of a special type of condensed vacuum spark.** J. Romand, G. Balloffet and B. Vodar (Centre Rech. Sci., Bellevue, Seine-et-Oise, France). *Spectrochim. Acta*, 1959, (6), 454–460 (in French).—The advantages of a three-electrode system in which the main analytical vacuum spark is triggered by an auxiliary spark are detailed. Results are given for the determination of O (0·02 to 1%) and N (0·002 to 0·06%) in titanium metal.
E. G. CUMMINS

1321. **Polarographic determination of antimony and copper in titanium dioxide in the analysis of rutile, anatase and brookite.** J. Stabryn (Moravské Chem. Závody, Ostrava, Czechoslovakia). *Hutn. Listy*, 1959, **14** (6), 515–516.—Titanium dioxide is converted with H_2SO_4 into $Ti(SO_4)_2$, the undissolved residue is filtered off and the Cu, Sb and part of the Pb are pptd. as sulphides; these are oxidised with HCl and Br, and after the removal of bromine are polarographed in a medium of HCl and NaH_2PO_2. *Procedure*—Dissolve the sample (5 g) in conc. H_2SO_4 (100 ml) and $Na_2SO_4.10H_2O$ (65 g) with heating, evaporate to 85 ml and cool till a glassy mass is formed. Add H_2SO_4 (1:3), dissolve and dilute to 800 ml. Filter, wash the filter with hot H_2SO_4 (1%) (100 ml), heat the filtrate to 80° and pass H_2S for 45 min. Filter off the sulphides, wash with aq. H_2S soln., oxidise on the filter with HCl and Br, and wash the filter with hot H_2O. Evaporate the filtrate to 40 ml, dilute to 50 ml with conc. HCl, transfer 20 ml to a 100-ml flask, add NaH_2PO_2 soln. (50%) (10 ml) and conc. HCl (50 ml), dilute to vol. and register the polarographic wave from 0 to −0·55 V. Well-defined waves are given by Sb (−0·10 V) and Cu (−0·30 V). Evaluate the results by the method of standard additions. The presence of NaH_2PO_2 ensures the reduction of Fe^{3+} to Fe^{2+}, and Cu^{2+} to Cu^{+}, and thus avoids the coincidence of the waves due to Fe and Cu.
J. ZÝKA

1322. **Tartrazine, a new selective reagent for zirconium.** G. Baiulescu and L. Turcu (Lab. for Inorg. and Anal. Chem., Univ. I. C. Parhon, Bucharest, Romania). *Anal. Chim. Acta*, 1959, **21** (1), 33–35 (in English).—Tartrazine (C.I. Acid Yellow 23) (**I**) quant. precipitates Zr^{IV} from soln. of pH $\not>$1. An excess of 2% **I** soln. is added to the sample soln., and heated to between 60° and 80°; the ppt. is filtered off and washed with water. The ppt. may be ignited to ZrO_2 or weighed as a complex having a factor of 0·32653 after drying at 110°. Sulphate, tartrate and nitrate interfere. Aluminium, Ti, UO_2^{2+}, La, Ce, K, Cd, Zn, Co, Ni, Cu, Mg, Na, Li, Fe and Ca give no reaction with **I** and the ppt. given by Ba, Th or Hg^{II} dissolve in acid.
T. R. ANDREW

1323. **Gravimetric estimation of zirconium by 3-ethylphenoxyacetic acid. I.** A. B. Sen and S. Misra (Chem. Dept., Lucknow Univ., India). *Z. anal. Chem.*, 1959, **168** (5), 343–347 (in English).—Zirconium is quant. pptd. with 3-ethylphenoxyacetic acid from HCl $\not>$0·15 *N*. The ppt. is of variable composition and is therefore ignited to the oxide before being weighed. None of the common cations interferes except Cr^{3+} and $V_2O_4^{2+}$, which are co-pptd. and can be removed by a double pptn. Up to a two-fold excess of SO_4^{2-} can be tolerated.
W. T. CARTER

1324. **Analytical applications of xylenol orange. II. Spectrophotometric study of the zirconium - xylenol orange complex.** K. L. Cheng (Kelsey, Hayes Co., New Hartford, N.Y., U.S.A.). *Talanta*, 1959, **2** (3), 266–269.—The purplish-red complex of Zr and xylenol orange (*Anal. Abstr.*, 1959, **6**, 4716; 2916) contains Zr and reagent in the ratio of 1:1. The max. extinction is at 535 mμ and the formation constant is 4×10^7. Xylenol orange can be used as an indicator in the titration of zirconium with EDTA.
W. J. BAKER

1325. **Determination of zirconium in zirconium driers. EDTA titration in 1 to 4 sulphuric acid.** C. F. Hirn and C. A. Lucchesi (Sherwin-Williams Co., Chicago, Ill., U.S.A.). *Anal. Chem.*, 1959, **31** (8), 1417–1418.—Zirconium in Zr naphthenate and octoate driers is rapidly determined (40 min.) by the addition of an excess of EDTA to a soln. of the sample in H_2SO_4 (1:4) and back-titration of the excess with $Bi(NO_3)_3$ soln., with thiourea as indicator. Zirconium (6%) in a 0·3-g sample is determined with a standard deviation of $\pm$0·03; the results agree well with those of the gravimetric mandelic acid method. *Procedure*—Dissolve the sample (0·3 g) in H_2SO_4 (1:4) (25 ml) and heat without boiling for 5 min. Add 10% aq. ammonium tartrate soln. (10 ml) and 0·05 *M* EDTA (disodium salt) (10 ml), adjust to pH 2 with aq. NH_3 (1:3), boil for 5 min., cool, and dilute to 100 ml. Add 1·3 g of thiourea and back-titrate with 0·05 *M* $Bi(NO_3)_3$ to the first yellow colour of the Bi - thiourea complex.
J. P. STERN

1326. **Photometric titration of thorium and the rare earths with (ethylenedinitrilo)tetra-acetic acid.** K. Y. Bril, S. Holzer and B. Réthy (Res. Lab., Orquima S.A., São Paulo, Brazil). *Anal. Chem.*, 1959, **31** (8), 1353–1357.—The rare-earth metals and Th are determined volumetrically with EDTA, with alizarin red S as indicator, or with photometric end-point detection at low concn. ($5 \times 10^{-6}M$). Direct and back-titration procedures are given for the determination of pure thorium soln., rare-earth-metal soln., and Th - rare-earth-metal mixtures. Thus 0·1% or 2% of Th in a mixture of rare-earth elements or 0·2% or 2% of rare-earth metals in thorium is determined with accuracies within $\pm$10% and 0·5%, or $\pm$10% and 5%, respectively. For the analysis of naturally occurring materials, a preliminary separation from interfering ions is achieved by the oxalate pptn. of Th and rare-earth metals in the presence of H_2O_2. Oxalate, Pb^{2+}, UO_2^{2+} and SO_4^{2-} do not interfere, but PO_4^{3-} and F^- do.
J. P. STERN

1327. **Analytical aspects of some azo dyes from chromotropic acid. VII. Spectrophotometric determination of thorium with nitroso-SNADNS.** S. K. Datta (Victoria Coll., Coochbehar, India). *Z. anal.*

Chem., 1959, **168** (5), 347–352 (in English).—Nitroso-SNADNS-4 [1:8-dihydroxy-7-nitroso-2:1′-azonaphthalene-3:6:4′-trisulphonic acid] forms a red complex with Th at pH 2·5. The max. extinction is at 520 mμ and the colour is stable for 24 hr. up to a temp. of 50°. Beer's law is obeyed over the range 0·4 to 60 p.p.m. and the sensitivity is 0·016 μg of Th per sq. cm. Some interference is caused by Sn, Fe^{II}, Ni and Co; Fe^{III}, Ce^{IV}, Zr, Au, PO_4^{3-} and F^- interfere seriously. The average error over the range 1·6 to 5·6 p.p.m. of Th is 1·14%. W. T. CARTER

1328. **Use of barbituric and 2-thiobarbituric acids for the quantitative determination of nitrite.** V. P. Krȳlov and N. S. Drozdov. *Khim. Nauka i Prom.*, 1958, **3** (6), 838–839; *Ref. Zhur., Khim.*, 1959, (16), Abstr. No. 56,887.—This spectrophotometric method is based on the reaction of NO_2^- with 2-thiobarbituric acid (**I**) to form 2-thiovioluric acid, with max. absorption at 570 mμ (pH 4·7). *Procedure*—To the test soln. add a 6- or 10-fold excess of phosphate - citrate buffer soln. (pH 4·7) and a 2 to 10-fold excess of 0·01 *M* **I**, heat for 15 min. at 100°, cool rapidly in ice water, and measure the extinction at 520 mμ. The colour is stable for >10 hr. Beer's law is not obeyed, but the deviations are reasonably small. The error is $\pm 5\%$ for concn. of NO_2^- $>10^{-4}$ *M*. No interference is caused by alkali metals, alkaline-earth metals, or NO_3^-. Barbituric acid is not suitable for the determination of NO_2^- because of the marked departure from Beer's law. C. D. KOPKIN

1329. **Determinations in inorganic analysis by extraction with the aid of coloured cations. III. Determinations by extraction with the aid of basic dyestuffs. C. Determination of traces of phosphate with safranine.** L. Ducret and M. Drouillas (Centre Nat. d'Études des Télécommunications, Dépt. P.C.M., Issy-les-Moulineaux, France). *Anal. Chim. Acta*, 1959, **21** (1), 86–90 (in French).—Molybdophosphates and tungstophosphates react with safranine (C.I. Basic Red 2) to give a complex, extractable by acetophenone - *o*-dichlorobenzene (3:1). *Procedure*—To 5 ml of neutral soln. add 0·1 *N* H_2SO_4 (10 ml), ammonium molybdate soln. (0·125%) (4 ml) and safranine soln. (0·2%) (1 ml) and mix. Set aside for 10 min., add 10 ml of solvent mixture, shake for 5 min., and record the extinction of the organic phase in a 1-cm cell at 532·5 mμ against a reagent blank prepared in the same way. The extinction decreases at the rate of 2% per hour. Arsenate, AsO_3^{3-}, VO_3^-, ClO_4^-, NO_3^-, SCN^-, $Cl_3C{\cdot}COO^-$, Au^{3+}, Bi^{3+}, Tl^{3+} and Sb^{3+} interfere. An extinction of 0·5 is given by 1 μg of P in a 1-cm cell. T. R. ANDREW

1330. **Spectrophotometric determination of mixtures of sodium pyrophosphate and sodium triphosphate.** J. Maurice (Lab. de Chim., École Nat. Sup. des Mines, Paris, France). *Bull. Soc. Chim. France*, 1959, (6), 819–822.—The bleaching of the $Fe(SCN)_3$ colour by pyro- and tri-phosphates is used to determine both species simultaneously. The effect of the two phosphates on the colour is markedly different at pH 0·4 and identical at pH 2·2. The effect of temp. and time on the absorption is investigated. *Procedure*—The sample of phosphate and 0·7 *M* KSCN (10 ml) are introduced into a 50-ml flask. A soln. (5 ml) of $(NH_4)_2SO_4.Fe_2(SO_4)_3$ (1 g per litre) made up in 4 *N* HNO_3 for the pH of 0·4 (after dilution), or in 0·058 *N* HNO_3 for the pH of 2·2, is added immediately before reading the exinction, and the mixture is diluted to 50 ml. From calibration curves, readings of the extinction at 470 mμ enable the total phosphate to be determined at pH 2·2 and the ratio of pyro- to triphosphate to be determined at pH 0·4. If the concn. is $\not> 1·5 \times 10^{-3}$ milli-equiv. at pH 2·2, and is $\simeq 3 \times 10^{-3}$, but $\not< 2 \times 10^{-3}$, milli-equiv. at pH 0·4, the accuracy is $\pm 3\%$. E. J. H. BIRCH

1331. **Solvent extraction of antimony(V) with ethyl acetate.** S. S. M. A. Khorasani and M. H. Khundkar (Dept. of Chem., Dacca Univ., E. Pakistan). *Anal. Chim. Acta*, 1959, **21** (1), 24–28 (in English).—From soln. 6 *N* with respect to HCl, Sb^{5+} can be extracted with ethyl acetate. The efficiency of extraction into an organic layer of final vol. 8 ml is 87% from a vol. of aq. soln. of 12 ml, the initial vol. of ethyl acetate and HCl soln. being equal. Ferric iron is completely extracted; Sn^{2+}, Cd^{2+} and Co^{2+} are partially extracted; large amounts of Cu^{2+} interfere. A procedure is proposed for the application of this technique to the analysis of antimonates and of type metal. The Sn in type metal must be oxidised to the quadrivalent state. T. R. ANDREW

1332. **Electro-analytical determination of bismuth.** P. N. Kovalenko. *Uch. Zap. Rostovsk. Univ.*, 1958, **40**, 139–148; *Ref. Zhur., Khim.*, 1959, (18), Abstr. No. 64,137.—It is shown that electrolytic pptn. methods for Bi in which the electrolyte is not stirred and the cathode potential is not controlled possess a number of disadvantages—the metallic ppt. are of poor quality, Bi is pptd. at the anode as an oxide as well as at the cathode, and the pptn. of small amounts of Bi takes a long time. The method for the rapid electrolytic pptn. of Bi with control of the cathode potential is considered to be an ideal method, apart from the complexity of the apparatus. This method gives insignificant deviations from the results of the gravimetric phosphate method. The separation of Bi from Cu by the use of complex-forming mixtures is completely satisfactory. C. D. KOPKIN

1333. **Spectrochemical determination of vanadium in alkali brines.** W. L. Baggett and H. P. Huyck (Pennsalt Chemicals Corp., Calvert City, Ky., U.S.A.). *Anal. Chem.*, 1959, **31** (8), 1320–1322.—The V (2 to 25 μg) in brine (1000 g) can be determined spectrographically on graphite electrodes coated with 20% NaOH soln. with a 2300-volt a.c. arc, the V being first concentrated by extraction of the 8-hydroxyquinoline complex into $CHCl_3$. The internal standard is Mo. The standard deviation on samples containing approx. 10 μg of V is $\pm 0·7$ μg. J. P. STERN

1334. **Determination of impurities in metallic vanadium. I.** V. V. Stepin, A. M. Pliss and E. V. Silaeva. *Byul. Nauch.-Tekh. Inform. Ural. Nauch. Inst. Chern. Metallov*, 1958, (4), 103–109; *Ref. Zhur., Khim.*, 1959, (15), Abstr. No. 53, 184.—A photometric determination is described of P in vanadium as the blue molybdophosphate complex after a preliminary separation of V on a cationite filter (H form). To increase the sensitivity of the method, the blue complex is extracted with butanol. Small amounts of P can be determined with an error of $\pm 0·0005\%$. Similar methods are described for silicon and arsenic. K. R. C.

1335. **Simultaneous spectrophotometric determination of niobium and tungsten. Application to complex alloy and stainless steels.** B. McDuffie, W. R. Bandi and L. M. Melnick (Appl. Res. Lab., U.S. Steel Corp., Monroeville, Pa.). *Anal. Chem.*, 1959, **31** (8), 1311–1315.—After separation of Nb, W, Ti, Ta and Mo as mixed oxides and the preliminary reduction of W^{VI} to W^{V} with $SnCl_2$ in HCl, Nb (0·08 to 3·0 mg) and W (0·07 to 4·0 mg) are determined together spectrophotometrically at 400 mμ as thiocyanates in 0·3 *M* KSCN - 20% (by vol.) acetone. The colour due to Nb is then destroyed by oxalate and W alone determined; Ta, Ti and Mo do not interfere. The relative error for Nb and W is generally <2·4% and 3·0%, respectively, but for some steels may be as high as 4% and 9%, respectively *Procedure*—Separate the mixed oxides of Nb, W, Ta, Ti and Mo by pptn. from acid soln. with cinchonine and ignition of the washed ppt. Fuse the oxides with 5 g (or 12·5 g) of $NaHSO_4$, dissolve the melt in 50 (or 125) ml of 15% tartaric acid soln. and dilute the cold soln. to 100 (or 250) ml. Reduce a 2 to 5-ml aliquot (diluted, if necessary to 5 ml) with 20 ml of 0·44 *M* $SnCl_2$ in 12 *N* HCl at 50° for 10 min., cool the soln., and add 20 ml of 0·75 *M* KSCN in 50% aq. acetone. Dilute to 50 ml and cool to room temp. in a thermostatically controlled bath; 15 min. after the addition of KSCN treat a portion of the soln. with 0·5 g of ammonium oxalate and shake. After 35 min. measure the extinction of the soln., both with and without the oxalate, against a blank or H_2O at 400 mμ. J. P. STERN

1336. **Determinations in inorganic analysis by extraction with the aid of coloured cations. III. Determinations by extraction with the aid of basic dyestuffs. D. Determination of traces of sulphate by methylene blue.** L. Ducret and M. Ratouis (Centre Nat. d'Études des Télécommunications, Dépt. P.C.M., Issy-les-Moulineaux, France). *Anal. Chim. Acta*, 1959, **21** (1), 91–94 (in French).—Sulphate ions are exchanged for an equivalent amount of SCN^- on Dowex 1-X4 resin (200 mesh, SCN- form). Thiocyanate is subsequently determined by extraction of its methylene blue complex at pH 1 into 1:2-dichloroethane. Sulphate ions in concn. as low as 10^{-6} *M* can be determined. T. R. ANDREW

1337. **Rapid determination of sulphur trioxide in aluminium sulphate.** H. Plaëtschke (Firma Gebr. Giulini G.m.b.H., Ludwigshafen, Germany). *Z. anal. Chem.*, 1959, **168** (4), 264–266 (in German).—Sulphate is determined by adding the soln. to an excess of 2 *N* $BaCl_2$ and back-titrating with *N* K_2SO_4, with tetrahydroxy-*p*-benzoquinone (disodium salt) (**I**) as indicator. The indirect method is necessary for samples containing Al because the Ba - **I** complex will not form in the presence of Al^{3+}. *Procedure*—To 15 ml of water add 3 drops of indicator soln. (0·13% of **I** in water containing 0·13% of KCl and 10% of $NaNO_2$). Add 2 *N* $BaCl_2$ (5 ml) and 5 ml of sample soln. containing 25 to 43 g of SO_4^{2-} per litre. Titrate to an orange end-point with *N* K_2SO_4. W. T. CARTER

1338. **Rapid colorimetric determination of selenium by the tin(II) - strong phosphoric acid reduction method.** Toshiyasu Kiba, Ikuko Akaza and Hiroaki Hachino (Chem. Dept., Kanazawa Univ., Japan). *Bull. Chem. Soc. Japan*, 1959, **32** (5), 454–458 (in English).—The method described is suitable for use with ores (*e.g.*, pyrites). The sample is decomposed with $SnCl_2$ - H_3PO_4 reagent (*cf.* Kiba *et al.*, *Anal. Abstr.*, 1958, **5**, 1874) in an atmosphere of hydrogen, and the hydrogen selenide liberated is absorbed in NaOH soln. and oxidised to selenic acid with H_2O_2; any hydrogen sulphide is simultaneously converted into H_2SO_4. Residual H_2O_2 is decomposed by boiling, the soln. is acidified with conc. HCl, then boiled to convert the selenic acid into selenous acid. This is reduced to colloidal elementary Se with $SnCl_2$ soln., and the extinction of the red colour is measured at 372 mμ. The recovery of Se is uniform even in the presence of large amounts of S and Fe. I. JONES

1339. **Rapid methods for potentiometric determination of tervalent chromium.** J. Allard, M. Bonnemay and C. Darrécamp (Lab. d'Electrolyse du CNRS). *Chrome Dur*, 1959–1960, 33–39.—Conditions are studied for the titration of Cr^{3+} with $KMnO_4$, especially in the presence of CrO_3. Platinum and calomel electrodes are used. The temp. is maintained at 50°. The effects of the concn. of Cr^{3+}, CrO_3 and $KMnO_4$ on the titration curves are described. N. E.

1340. **Influence of the total composition of rocks on the results of the spectrographic determination of molybdenum in granitoids.** É. E. Vaĭnshteĭn and L. I. Pavlenko. *Fiz. Sb. L'vovsk. Univ.*, 1958, [4 (9)], 120–123; *Ref. Zhur., Khim.*, 1959, (18), Abstr. 64,120.—The calibration curves of log $(I_{Mo}/I_{background})$ *vs.* log *C*, relating to matrices of granite, granodiorite or diorite and constructed by exciting the spectra in an a.c. arc with evaporation from a carbon electrode, are displaced relatively to each other. By using ^{99}Mo, evaporation curves and excitation curves were obtained separately. By measuring the β-activity of the residues in the electrode craters after the exposure, the number of atoms evaporated (in %) in various times was calculated. The intensity of the line Mo 3170·3 Å was determined by using moving plates. It was shown that the evaporation curves exhibit horizontal sections, owing to a reduction in the rate of evaporation of Mo after the first few tens of seconds of burning of the arc. The rate of evaporation then increases again, but the moment at which this takes place varies with different rocks. The conditions for the excitation of the atoms of Mo were, however, identical for all rocks; this was confirmed by the const. temp. of the discharge. The reason for the differing evaporation of Mo is the influence of Ca and Mg which, when evaporated from various rocks, carry Mo with them to different extents. The relationship between the displacement of the curve and the concn. of these elements is linear. C. D. KOPKIN

1341. **Nicotinamidoxime as an analytical reagent. I. Spectrophotometric determination of uranium with nicotinamidoxime.** K. K. Tripathi and D. Banerjea (Dept. of Inorg. and Anal. Chem., Indian Ass. for Cultivation of Science, Jadavpur, Calcutta). *Z. anal. Chem.*, 1959, **168** (5), 326–329 (in English).—Sexavalent U gives a yellow complex with nicotinamidoxime at pH 10·9 to 11·5. The colour is stable for at least 1 hr., obeys Beer's law over the range 5 to 40 p.p.m. and has a sensitivity of 0·045 μg per sq. cm. The common anions are without effect although PO_4^{3-}, CO_3^{2-} and CN^- interfere seriously. Interference from cations can be suppressed by tartrate or EDTA, but Cu, Fe and V must be absent. W. T. CARTER

1342. **Rapid methods for the analysis of metals and crude minerals. VIII. The determination of small amounts of uranium in crude minerals.** Z. Šulcek, J. Michal and J. Doležal (Central Inst. Geol., Charles' Univ., Prague). *Coll. Czech. Chem. Commun.*, 1959, **24** (6), 1815–1821 (in German).—The U is adsorbed on silica gel, eluted with conc. HCl and determined polarographically. *Procedure*—Prepare a soln. of the ore containing $\not<$0·3 mg of U. Acidify with conc. HCl, evaporate to dryness, add conc. HCl (2 ml), dilute with warm water and filter. To the filtrate add tartaric acid (3 g) and EDTA disodium salt) (0·2 *M*) (20 to 30 ml), boil, cool and add aq. NH_3 - $(NH_4)_2SO_4$ soln. [132·15 g of $(NH_4)_2SO_4$ and 875 ml of 10% aq. NH_3 diluted to 1 litre] (25 ml). Filter slowly through a column of silica gel and wash with water (100 ml). Elute the U with conc. HCl followed by several 5-ml portions of water. Evaporate to dryness, add conc. HCl (0·5 ml), dilute with water, add ascorbic acid (0·2 g) and neutralise with NaOH. Add $HClO_4$ (70%) (1·25 ml) and thymol soln. (0·075%) (0·5 ml) and dilute to 25 ml. Remove dissolved O with N and determine the U polarographically. For a series of 8 samples of known composition containing 0·078 to 8·19% of U the error varied from −0·11 to +0·05%. W. T. CARTER

1343. **Polarographic determination of sexavalent uranium in uranium oxides; determination of oxygen to uranium ratios.** R. M. Burd and G. W. Goward (Westinghouse Electric Corp., Bettis Plant, Pittsburgh). *U.S. Atomic Energy Comm.*, Rep. WAPD-205, 1959, 11 pp.—Oxide samples are dissolved in hot conc. H_3PO_4, and the U^{VI} is determined polarographically in a H_2SO_4 - H_3PO_4 medium with a dropping mercury electrode. The method provides an accurate means for determining O to U ratios in high-purity nominal uranium dioxide, and has been applied to the analysis of raw uranium dioxide, fuel pellets used in the PWR Core 1 blanket, and a variety of oxides prepared for development work on ceramic fuel materials. The relative precision of the method at the 95% confidence level is ±4%, and the range of the method is from 0·2 to 20·0% of U^{VI} in high-purity uranium oxides. NUCL. SCI. ABSTR.

1344. **Polarography of uranium(IV) in non-complexing and complexing media. Amperometric determination of fluoride.** D. J. McEwen and T. De Vries (Dept. of Chem., Purdue Univ., Lafayette, Ind., U.S.A.). *Anal. Chem.*, 1959, **31** (8), 1347–1351.—In an investigation of the polarography of U^{IV} in non-complexing (perchlorate, chloride and sulphanilate-buffered) media and in complexing (formate, acetate and chloroacetate) media, it was found that the anodic wave of U in formate or chloroacetate buffer (pH 3·5 to 3·7) is suitable for the titrimetric determination of F^-; 0·04 to 2·2 mg of F^- can thus be determined with an accuracy better than ±10%. *Procedure*—De-oxygenate 20 ml of 0·2 *M* formate buffer (pH 3·5 to 3·7) and the F^- soln., mixed in a polarography cell, by bubbling with N. Titrate with 0·012 *M* UCl_4, allowing the soln. to equilibrate and taking current readings after each addition. J. P. STERN

1345. **Sensitive reaction for the detection of bromate.** E. Ružička (Chem. Inst., Pädagog. Hochsch., Olomouc). *Coll. Czech. Chem. Commun.*, 1959, **24** (6), 2062–2064 (in German).—Bromate in 0·5 *N* HCl soln. can be detected by a colour reaction with 3-aminophenoxazin-2-one (**I**). The presence of ClO_3^-, IO_3^-, Cl and Br can be tolerated, but ClO^-, BrO^-, NO_2^-, H_2O_2, $S_2O_8^{2-}$, and iodine interfere. *Procedure*—To 2 ml of a soln. of BrO_3^- in 0·5 *N* HCl add 0·2 ml of **I** soln. (0·0001 *M* in ethanol). A yellow colour indicates the presence of BrO_3^-. Chlorate and IO_3^- give positive reactions in 6 *N* HCl soln. W. T. CARTER

1346. **Influence of iron on the colorimetric determination of manganese.** R. K. Korabel'nik. *Nauch. Dokl. Vÿssh. Shkolÿ, Metallurgiya*, 1959, (1), 250–253; *Ref. Zhur., Khim.*, 1959, (17), Abstr. No. 60,514.—It is shown by photometry of soln. of $KMnO_4$ and by potentiometric titration of soln. of $KMnO_4$ with a soln. of $FeSO_4$ in the absence and presence of Fe^{3+}, that Fe^{3+} form complexes with permanganic acid, causing errors in the photometric determination of Mn in steel as MnO_4^-. It is shown that the conversion of the Fe^{3+} into the more stable complex with H_3PO_4 prevents the formation of the permanganate complex and eliminates the errors. With an excess of H_3PO_4 the formation of complexes between permanganic and phosphoric acids was also observed; this stabilises the MnO_4^- in soln. and widens the limits of applicability of the photometric method. C. D. KOPKIN

1347. **Spectrographic determination of iron, aluminium, calcium, magnesium, copper and nickel in high-purity metallic manganese.** V. F. Erko and N. I. Bugaeva. *Fiz. Sb. L'vovsk. Univ.*, 1958, [4 (9)], 490–491; *Ref. Zhur., Khim.*, 1959, (18), Abstr. No. 64,162.—The sample is dissolved in HNO_3, the soln. evaporated, and the residue ignited at 200°. The resulting powder is placed in a cavity in a carbon electrode and evaporated in a d.c. arc at 13 amp., with an arc gap of 4·5 mm and exposure for 90 sec. The spectra are photographed in a medium spectrograph on plates of sensitivity 22 GOST units. The analysis is carried out for concn. of impurities from 0·003 to 0·1%, from calibration curves of log *I* *vs.* log *C*, allowing for the background. Standards are prepared from a soln. of $Mn(NO_3)_2$, obtained from $KMnO_4$. The impurities are added as soln., with subsequent pptn. of hydroxides, and ignition. The error of the analysis is ±20%. C. D. KOPKIN

1348. **Determination of rhenium in molybdenites and industrial wastes.** O. A. Suvorova and F. G. Karinskaya. *Trudÿ Inst. Metallurgii i Obogashcheniya, Akad. Nauk KazSSR*, 1959, **1**, 142–151; *Ref. Zhur., Khim.*, 1959, (16), Abstr. No. 56,873.—This rapid and simple procedure evolved for determining small amounts of Re is based on the separation of Re from most other elements by sintering with CaO, followed by extraction of the sintered product with water and subsequent photometry of the Re in HCl soln. as the thiocyanate complex. *Procedure*—Grind the finely powdered sample (2 to 5 g) in a mortar with 2 to 4 times the amount of CaO, and ignite the mixture at 500° to 700° for 4 hr. with periodic mixing. Cool, and extract with hot water (3 × 40 to 50 ml), filtering each extract. Wash the residue with water (3 × 20 to 30 ml) and evaporate the combined filtrate and washings (250 to 300 ml) to 5 or 10 ml (with a concn. of Re >0·10 mg per ml evaporation is not necessary). Transfer the soln. and pptd. calcium salts to a 10- or 25-ml flask, make up to vol. and allow to settle. Transfer 1 to 5 ml (depending on the concn. of Re) of the supernatant liquid to a colorimeter tube, add 2 or 3 ml of conc. HCl, 0·5 ml of 20% NH_4SCN

soln. and 1 ml of a 20% soln. of $SnCl_2$ in conc. HCl; dilute to 10 ml, set aside for 30 or 40 min., and compare the colour with that obtained from standard rhenium soln. (0·5 to 1·2 ml, containing 0·03 mg of Re per ml). The error is 5 to 10%; the sensitivity is 0·0001 mg of Re per ml.

C. D. KOPKIN

1349. **Ferrous metallurgical analysis.** B. Bagshawe (The Brown-Firth Res. Lab., Princess St., Sheffield, England). *Analyst*, 1959, **84**, 475–504.—Methods for the determination of 25 elements that may occur in steel, either as alloying constituents or as native impurities, are reviewed and discussed. These elements are Al, As, B, C, Cr, Co, Cu, H, Pb, Mg, Mn, Mo, Ni, Nb, Ta, N, O, P, Si, S, Sn, Ti, W, V and Zr. (188 references.)

A. O. JONES

1350. **Spectrophotometric determination of iron with 3-hydroxy-2-naphthoic acid.** A. K. Majumdar and C. P. Savariar (Dept. of Inorg. and Anal. Chem., Jadavpur Univ., Calcutta, India). *Anal. Chim. Acta*, 1959, **21** (1), 47–53 (in English).—3-Hydroxy-2-naphthoic acid (**I**) reacts with Fe^{3+} in acid soln. to give a blue water-soluble complex. The excess of **I** is insoluble, but pptn. is prevented by the addition of gelatin. The pH of the soln. should be between 2·9 and 3·1. The max. absorption is at 590 mμ (extinction = 1·0 for 30 p.p.m. of Fe^{III} in a 1-cm cell). The colour is stable for almost 1 hr. The interference of 21 commonly occurring ions is reported.

T. R. ANDREW

1351. **Photometric determination of ferric iron by catechol.** A. N. Smith (Res. Inst. of E. Africa, Kericho, Kenya, E. Africa). *Analyst*, 1959, **84**, 516.—The soln. of the sample in acid, neutralised with NaOH to the yellow end-point of *p*-nitrophenol indicator, is made slightly alkaline, a catechol soln. and a buffer soln. are added and, after 1 hr., the extinction of the cherry-red liquid is measured at 580 mμ and compared with that of standard soln. Beer's law is obeyed for concn. of Fe up to 15 p.p.m. The method is not applicable in the presence of V. The accuracy is satisfactory in the presence of Cu, Ni, Co, Mn and W.

A. O. JONES

1352. **Estimation of microgram quantities of iron in culture medium, using bathophenanthroline.** P. A. Seamer (Wellcome Res. Lab., Langley Court, Beckenham, Kent, England). *Nature*, 1959, **184** (Suppl. No. 9), 636–637.—The sample is digested with H_2SO_4, HNO_3 and $HClO_4$ and the colour subsequently formed with the reagent is extracted into *n*-hexanol for measurement at 533 mμ. The use of this reagent is based on the findings of Smith *et al.* (*Analyst*, 1952, **77**, 418).

H. F. W. KIRKPATRICK

1353. **Spectrophotometric determination of iron in titanium dioxide.** W. Palatzky. *Silikattech.*, 1959, **10**, 133.—Experiments showed that, when TiO_2 is heated with HCl, traces of iron in the TiO_2 do not dissolve completely; the photometric method therefore gives low results. It is better to dissolve TiO_2 completely in H_2SO_4 and to use the thiocyanate method for determining Fe (accuracy $\pm$0·7%).

BRIT. CERAM. SOC. ABSTR.

1354. **Determination of iron in uranium dioxide.** U.K.A.E.A. (Res. Group, Chatham Outstation, Kent). AERE-AM 36, 1959, 3 pp.—The sample is dissolved in 50% HNO_3, any insol. residue is fused with $KHSO_4$ and the melt is mixed with water and filtered into the main soln. The Fe is converted into its thioglycollate and determined absorptiometrically. Uranium is complexed with ammonium citrate, and ammonium carbonate is added to suppress the colour formed with thioglycollic acid. Interference from NO_2^- is eliminated by the addition of sulphamic acid. The method is suitable for Fe contents $\not>$350 p.p.m. and errors should not exceed $\pm$5%, except in the lowest ranges. (This method is adapted from the Springfields Method No. 50A and was written in its present form by J. Walkden and K. E. Heathfield.)

G. S. ROBERTS

1355. **Determination of iron in low-grade uranium ores.** U.K.A.E.A. (Res. Group, Woolwich Outstation, Royal Arsenal, Woolwich, London). AERE-AM 59, 1959, 2 pp.—The method is based on standard practice and has been written with full details by A. Parker. It has been used for the analysis of uranium-bearing ores containing 5 to 40% of Fe.

1356. **Iron in ores, slags and refractories.** British Standards Institution (2 Park St., London). Amendment No. 3 to B.S. 1121: Part 33: 1955 (published 23.6.59).—A revised calibration table is given.

1357. **Radial chromatography in the analysis of iron oxides and ferrites containing various impurities.** E. Bovalini and M. Piazzi (Univ. Siena, Italy). *Ann. Chim., Roma*, 1959, **49** (5–6), 1075–1081.—Radial chromatography, as used in the qual. and quant. analysis of aluminium and steel alloys (*cf.* Venturello and Ghe, *Brit. Abstr. C*, 1953, 54; *Anal. Abstr.*, 1955, **2**, 2100), is applied to the analysis of iron oxides and ferrites of mineral origin, obtained by pptn. and calcination of hydroxides. Small amounts of Al, Mn, Cu, Co, Ni, Ti and Zn can thus be separated and determined. The min. limits of detection and determination of each element are determined, and a detailed procedure is given.

C. A. FINCH

1358. **Flame-photometric determination of iron, copper and cobalt in cobalt mattes and concentrates.** N. McN. Galloway (Chem. Lab., Roan Antelope Copper Mines Ltd., Luanshya, N. Rhodesia). *Analyst*, 1959, **84**, 505–508.—The soln. of the sample in HCl, oxidised with HNO_3 and Br to remove S, is evaporated with HCl. The filtered soln. of the residue in HCl is extracted with ethyl methyl ketone - $CHCl_3$ (2 : 1) and then with $CHCl_3$. After removal of the solvent the extract is oxidised with H_2O_2 and H_2SO_4 until free from C. An aq. soln. of the residue, suitably diluted, is used for the determination of Fe. The aq. layer from the extraction, concentrated after addition of HNO_3 and diluted with specified amounts of water and *iso*propyl alcohol, is used for the determination of Co, and a further dilution for the determination of Cu. Determinations are made with the Zeiss PMQ11 quartz spectrophotometer with flame attachment. Agreement with the results of chemical determinations is good.

A. O. JONES

1359. **Spectrographic determination of residual elements in steel. Report No. 2. The excitation index technique.** B.I.S.R.A. Spectrographic Analysis Sub-Committee. *J. Iron St. Inst.*, 1959, **192** (3), 253–256.—The investigation described proved that a good measure of standardisation could be achieved

with widely differing source units. The excitation conditions were specified by means of the intensity ratios of a non-homologous line-pair of the major element present. The determination of contents down to 0·01% was achieved on most of the residual elements tested, except W (0·05%). A mean coefficient of variation of ±4·2% was achieved.
J. W. O. Pyemont

1360. **Spectrographic determination of the phase content of aluminium in steel.** R. E. Levitin and V. I. Smirnova. *Fiz. Sb. L'vovsk. Univ.*, 1958, [4(9)], 497–501; *Ref. Zhur., Khim.*, 1959, (18), Abstr. No. 64,110.—To determine the total content of Al the spectra are excited in an a.c. arc at 5 amp. with a copper electrode sharpened to a cone. The spectra are photographed in a medium spectrograph with ignition for 10 sec. and exposure for 20 sec. with an arc gap of 1 mm and slit width 0·015 mm. The analysis is carried out with the lines Al 3082·1 and Fe 3055·26 A. To determine metallic aluminium only, in the concn. limits 0·01 to 0·2%, the sample (1 g) is dissolved in HCl (1:1) (10 ml), the ppt. of Al_2O_3 is filtered off, and the soln. is treated with conc. HNO_3 to oxidise Fe. Synthetic samples are prepared by dissolving Armco iron and adding $AlCl_3$. Both carbon electrodes are steeped in the test soln. and dried for 30 min. at 400°, and the spectra are excited at 5 amp. with an arc gap of 1 mm; the ends of the electrodes are shaped to hemispheres; the slit is illuminated by the light from the central portion of the arc by means of a diaphragm of diam. 3 mm. The ignition curves are studied. Calibration curves are constructed. The times of ignition and exposure are not stated. The mean error of one determination is $\simeq \pm 9\%$.
C. D. Kopkin

1361. **Spectrochemical determination of acid-soluble aluminium in low-alloy steels by the rotating-disc method.** F. G. Howell (Watertown Arsenal Lab., Mass.). *U.S. Atomic Energy Comm.*, Rep. WAL-TR-804/19, 1959, 18 pp.—A solution technique with a rotating graphite disc and a high-voltage a.c. spark was used. The method is rapid compared with chemical analysis. Of the common elements used in low-alloy steels, only W interferes. The method may be applied to samples containing 0·02 to 0·25% of acid-soluble aluminium.
Nucl. Sci. Abstr.

1362. **Aluminium in iron, steel and ferro-alloys.** British Standards Institution (2 Park St., London). Amendment No. 1 to B.S. 1121: Part 35: 1955 (published 23.6.59), 2 pp.—Amendments to the reagents and analytical methods are given.

1363. **Acidimetric determination of silicon precipitated as potassium fluorosilicate applied to the rapid determination of silicon in ordinary steel, stainless (nickel - chrome) steel, tungsten steel and cast iron.** P. Jehenson, E. Buslin and C. Vandall. *Ind. Chim. Belge*, 1959, **24** (4), 341–348.—The method described previously (*Ibid.*, C.R. 27th Congress of Ind. Chem., 1954, **20**, Spec. No. Vol. II, 453) for the determination of Si in ordinary steel has been successfully applied to stainless steel, tungsten steel and cast iron. Suitable proportions of HCl and HNO_3 for the rapid dissolution of stainless steel have been investigated, *e.g.*, for a 3-g sample of steel, water (30 ml), conc. HCl (d = 1·19) (15 ml) and conc. HNO_3 (d = 1·40) (15 ml) are employed. For dissolving tungsten steel, H_3PO_4 is used in addition, and the determination is made without previous separation. The results are in good agreement with those obtained by the standard method of dehydration by evaporation to white fumes, and a systematic error of $\simeq$0·03% is observed in the method of dehydration by $HClO_4$.
J. M. Jacobs

1364. **Spectrographic determination of titanium in steels of the types Kh18N9T and EI-123.** M. Z. Nesanelis. *Fiz. Sb. L'vovsk. Univ.*, 1958, [4(9)], 406–410; *Ref. Zhur., Khim.*, 1959, (18), Abstr. No. 64,117.—The spectra are excited in an a.c. arc at 3 amp., with an arc gap of 1·5 mm, a secondary gap of 0·8 mm, and a current in the transformer circuit of 0·1 amp. A copper electrode sharpened to a truncated cone with an end-surface of diam. 1·5 mm is used. The spectra are photographed in a medium spectrograph ISP-22 with ignition for 10 sec. and exposure for 30 sec. The analysis is carried out from the lines Ti 3088·0 and Fe 3083·37 A. The slit, of width 0·02 mm, is illuminated by using a three-lens system with a 5-mm diaphragm. The coeff. of variation is ±3·3%. It is demonstrated experimentally that the structure of the samples has no influence on the accuracy of the analysis. With a content of 2% of W in the steel, the calibration curve is displaced; by allowing for the displacement by the parallel-curve method, steels of type EI-123 can be analysed with satisfactory accuracy.
C. D. Kopkin

1365. **Determination of nitrogen in carbon steel.** E. Penna (Lab. da CSN, Volta Redonda, Rio de Janeiro). *ABM, Bol. Ass. Brasil Metais, São Paulo*, 1959, **15** (56), 449–458.—The steel is dissolved in dil. H_2SO_4 (30%, by vol.) and heated to fuming to convert the nitride N into ammonium salts; the soln. is diluted with water, and made alkaline with NaOH, and the NH_3 is distilled off and determined volumetrically, or colorimetrically with Nessler reagent.
L. A. O'Neill

1366. **Determination of oxygen in steel.** E. Penna (Lab. da CSN, Volta Redonda, Rio de Janeiro). *ABM, Bol. Ass. Brasil Metais, São Paulo*, 1959, **15** (56), 459–465.—The molten steel is treated with metallic aluminium to convert the oxides present into Al_2O_3. The product is then dissolved in HCl (1:1), HNO_3 is added and the Al_2O_3 content of the residue is determined by conventional methods after fusion with $K_2S_2O_7$.
L. A. O'Neill

1367. **Nephelometric determination of oxygen in steel.** M. Šícha (VŽKG, Ostrava, Czechoslovakia). *Hutn. Listy*, 1959, **14** (6), 513–515.—The method is based on the reaction of metallic aluminium with the molten sample and the turbidimetric measurement of the Al_2O_3 formed after dissolution of the product in acid. The interference of alloying elements (*e.g.*, Ni, Cr, Mo and V) which impart colour to the soln. is allowed for by centrifuging the comparison soln. and by measuring the turbidity by means of a photometer or nephelometer. The procedure occupies <25 min. *Procedure*—Weigh 2 g of filings of the sample (which has been fused with 1% of aluminium) into each of two beakers, dissolve in conc. HNO_3 - conc. H_3PO_4 - H_2O (9:3:8) (30 ml), warm, add $(NH_4)_2S_2O_8$ soln. (10%) (10 ml) and boil for 13 min. till the persulphate is decomposed. Cool and dilute the soln. to 50 ml with HNO_3 (4%). Separate the Al_2O_3 in one sample by centrifuging and compare both soln. in a Spekker photometer with the use of Ilford filter No. 508. Nickel (<3%), Mo (<0·4%) and Cr (<0·7%) do not interfere.
J. Zýka

1368. **Determination of oxygen from the content of non-metallic inclusions in rimming steel.** A. Šmrhová and J. Janáček (VŽKG, Ostrava, Czechoslovakia). *Hutn. Listy*, 1959, **14** (6), 559–564.—A critical survey of known methods is given and the results obtained by a method based on electrolytic isolation followed by the extraction of individual components of the inclusions by means of various chemical reagents are discussed. The following electrolyte has been used for isolation—Na citrate (150 g) and KBr (12 g), per litre of H_2O, as anolyte and citric acid (86 g) and N Na_2CO_3 (1 ml), per litre of H_2O, as catholyte. The pH (9) of the electrolyte must be carefully maintained throughout the electrolysis. It has been found in comparative tests that the total content of O calculated from the content of inclusions agrees well with that by the vacuum extraction method for the outer parts of the ingot (a sample of mild rimming steel), but the result by the vacuum extraction method was higher for the central portion. The average error of a single determination by the present procedure is $\pm 10\%$. J. Zýka

1369. **Determination of sulphur in iron and steel.** C. L. Luke (Bell Telephone Lab. Inc., Murray Hill, N.J., U.S.A.). *Anal. Chem.*, 1959, **31** (8), 1393–1394.—Sulphur (0·0005 to 0·05%) in iron or steel is rapidly determined by absorption of the H_2S (liberated from an acid soln. of the sample by a HI - H_3PO_2 mixture) in an excess of ammoniacal $ZnCl_2$ soln. This soln. is then titrated with IO_3^- soln. standardised against an iron or steel sample of known S content. The accuracy is within ±20%. J. P. Stern

1370. **Spectrographic determination of sulphur in iron-base alloys.** A. M. Kripskiĭ. *Sb. Dokl. I. Nauch.-Tekh. Konf. Spektr. Anal., Minsk*, 1956, 22–23; *Ref. Zhur., Khim.*, 1959, (15), Abstr. No. 53,161.—Standards are prepared with a sulphur concn. of 0·02 to 26%. The best sources are a high-voltage condensed spark and a high-frequency spark. About 50 lines were excited, the most sensitive being at 5454 A, but sensitivity is lost at concn. of $<3\%$. The intensity of the lines was greater with minimum inductance of the discharge circuit ($<20\ \mu H$). Atmospheric N was used for the comparison line (*i.e.*, 5679 A). In the determination of 0·01 to 0·2% of S in pig-iron and steel, the electrodes were used in an atmosphere at 50 to 80 torr. Sulphur spectra were excited in the high-voltage spark up to a concn. of 0·009% with little background. The presence of S in the samples considerably augmented the intensity of the Fe lines; this increase was independent of the concn. of the substance entering the discharge. An increase in the intensity of S lines was also observed with an increased concn. of Fe. K. R. C.

1371. **Determination of the uranium content of the National Bureau of Standards iron and steel chemical standards.** G. A. Welford and D. Sutton (New York Operations Office, Health and Safety Lab., AEC). *U.S. Atomic Energy Comm.*, Rep. NYO-4755, 1957, 26 pp.—A procedure for the isolation of uranium from iron and steel matrices is outlined, and has been used to determine the U content of ten N.B.S. iron and steel chemical standards. The U is adsorbed from a soln. of the iron or steel standard in N H_2SO_4 on to Amberlite IRA-400 (SO_4^{2-} form); interfering cations are washed from the resin with 0·05 N HCl and the U is then eluted with 2 M $HClO_4$. Any residual Fe, usually <5 mg, is removed from the uranium fraction by mercury cathode electrolysis or by cupferron extraction. The resulting soln. is analysed photofluorimetrically. The recovery is $>90\%$ when 0·5 to 5 μg of U is added to soln. containing 5 g of iron or steel. Nucl. Sci. Abstr.

1372. **Spectrographic analysis of cobaltic oxide for impurities.** G. P. Skornyakov, Z. A. Motova, T. P. Chukina, A. R. Romashenko and A. T. Novgorodtseva. Symposium: Reports of the First Urals Convention on Spectroscopy, 1956. Sverdlovsk, Metallurgizdat, 1958, 62–63; *Ref. Zhur., Khim.*, 1959, (16), Abstr. No. 56,927.—The sample is placed in a cavity (diam. 1·5 mm, depth 2 mm) in a carbon electrode the end of which is sharpened to a cone; the upper electrode is sharpened to a truncated cone. The spectra are excited in an a.c. arc discharge at 7 amp. with ignition for 30 sec. and exposure for 90 sec., and are photographed in a large spectrograph. The determinable concn. are—Ni and Fe 0·1 to 1·0%, and Cu and Mn 0·01 to 0·4%. The analytical lines (in A) are Ni 3099·1 and Co 3054·7, Fe 2719·0 and Co 2719·5, Mn 2801·0 and Co 2803·7, and Cu 2997·3 and Co 3048·1. Standards are prepared from oxides obtained by igniting suitable salts. C. D. Kopkin

1373. **Spectrographic analysis of cobalt for metallic impurities, with cast electrodes.** E. E. Belokrinitskaya, V. V. Bondarenko, I. N. Vitushkina, M. S. Gerasimova, V. L. Ginzburg, I. N. Gramenitskiĭ, D. M. Livshits and V. F. Kryzhnaya. Symposium: Reports of the First Urals Convention on Spectroscopy, 1956. Sverdlovsk, Metallurgizdat, 1958, 59–61; *Ref. Zhur., Khim.*, 1959, (16), Abstr. No. 56,920.—The samples are cast into rods of diam. 7 mm and length 40 mm. The ends of the rods are ground to a flat surface and treated with HCl (1:1) for purification from Fe. The spectra are excited in an a.c. arc with an upper carbon electrode and photographed in a medium quartz spectrograph. The standards are prepared from pure cobalt, and the impurities are determined chemically. The method can be used to determine Ni, Fe, Si, Mn, Al, Cu, As and Sb with a mean error of 5 to 15%. C. D. Kopkin

1374. **Amperometric titrations with a rotating platinum electrode. V. Determination of the cobalt content of metals, alloys and silicates with potassium ferricyanide.** I. Bozsai (Chem. Lab., Central Material Examining Dept., Csepel Iron- and Metal-works, Budapest). *Magyar Kém. Foly.*, 1959, **65** (6), 207–211.—The Co^{II} in 100 ml of sample soln. also containing tartaric acid soln. (30%) (10 ml) and aq. NH_3 (sp. gr. 0·91, 50 ml) is oxidised with $\simeq 0.033$ M $K_3Fe(CN)_6$ (5 ml). The excess of $K_3Fe(CN)_6$ is back-titrated with a standard soln. of $CoCl_2$, which contains 2 mg of Co^{II} per ml (prep. described). The end-point is observed with a rotating platinum micro-electrode, which is polarised to 0·00 V *vs.* the S.C.E. The following ions do not interfere—Fe^{3+}, Ni^{2+}, Cr^{3+}, Al^{3+}, Zn^{2+}, Mg^{2+}, Cu^{2+}, Cl^-, SO_4^{2-}, PO_4^{3-}, NO_3^-, ClO_4^-, MoO_4^{2-}, VO_3^- and $Cr_2O_7^{2-}$; Mn^{2+} interfere as they are titrated simultaneously with Co^{2+}. Reducing and oxidising agents, and many other ions, interfere. The error of the determination is $\pm 5\%$ for 0·01 mg of Co^{2+} per 100 ml of soln. and can be reduced by using a more dilute soln. of $CoCl_2$. A. G. Peto

1375. **Colorimetric determination of nickel with dimethylglyoxime.** Z. Gregorowicz (Inst. f. anorg. Chem., Schlesischen Tech. Hochsch., Gliwice, Poland). *Z. anal. Chem.*, 1959, **168** (4), 241–246 (in German).—In the determination of Ni with dimethylglyoxime and satd. aq. iodine soln., no interference is caused by up to a 40-fold excess of Fe^{3+} if K Na tartrate is added and the pH of the final soln. is >8. Up to a 100-fold excess of Al^{3+}, Ca^{2+}, Mg^{2+}, Cl^-, NO_3^-, and SO_4^{2-} is without effect.
W. T. Carter

1376. **Estimation of nickel by αβ-dioximino-butyranilide.** J. S. Dave and A. M. Talati (M.S. Univ. of Baroda, India). *J. Indian Chem. Soc.*, 1959, **36** (5), 302–304.—αβ-Dioximinobutyranilide precipitates Ni quantitatively as $Ni(C_{10}H_{10}O_3N_3)_2$ at pH 6·2 to 8·6; Br^-, I^-, SO_4^{2-}, Hg^{2+}, Cd^{2+}, Zn^{2+}, Ca^{2+}, Ba^{2+} and Mg^{2+} (and Cr^{3+} and Fe^{3+} after complexing with citric or tartaric acid) do not interfere.
I. Jones

1377. **Critical evaluation of spectrographic, fluorescence X-ray, and polarographic methods for the determination of the platinum metals.** F. E. Beamish (Univ., Toronto, Ontario, Canada). *Talanta*, 1959, **2** (3), 244–265.—A review of the literature up to June, 1958, is presented, with an evaluation of methods for assaying silver beads and lead buttons (obtained by fire assay), determining the purity of ppt. of platinum metals, and analysing platinum alloys free from or containing base metals. The max. accuracy in indirect spectrographic determinations of Pt, Pd and Rh depends largely on the attainment of uniformity of composition of button, bead, ppt. or alloy, and on the production of standards having identical metallurgical histories. Osmium, Ir and Ru in ores, concentrates and alloys cannot be accurately determined, either directly or indirectly, by spectrography. Under optimum conditions, the accuracies of spectrographic and of wet methods are comparable, but spectrography is superior for the qual. analysis of ppt. containing trace amounts of other metals, or for the micro-determination of platinum metals in an alloy. Polarographic methods are at present restricted to samples (not assay buttons or beads) containing a few elements in low concn. For the rapid analysis of many routine samples of similar composition, spectrographic or polarographic methods are preferable. Published X-ray fluorescence methods are too few in number to allow their relative accuracy and precision to be assessed. (68 references.)
W. J. Baker

1378. **Spectrophotometric determination of ruthenium with NN′-bis-(3-dimethylaminopropyl)-dithio-oxamide.** W. D. Jacobs and J. H. Yoe (Dept. of Chem., Univ. of Virginia, Charlottesville, U.S.A.). *Talanta*, 1959, **2** (3), 270–274.—From 15 to 178 μg of Ru can be determined after an initial separation from interfering ions (Rh^{3+}, Ir^{4+}, Pt^{4+}, Pd^{2+}, osmate) by distillation as RuO_4, which is collected in 6 *M* HCl. *Procedure*—Evaporate the distillate (10 ml) to ≃1 ml, transfer it to a 25-ml flask with a few ml of H_2O, add conc. HCl (10 ml) and the reagent soln. (0·087% in 95% ethanol) (3 ml). Heat the mixture in a boiling-water bath for 15 min., cool, and make up to the mark with H_2O. Measure the extinction of the soluble blue Ru complex at 630 mμ against a water blank (1-cm matched Corex cells). The sensitivity is 0·02 μg per sq. cm and the standard deviation is 0·06 p.p.m. of Ru (10 determinations). This new Ru complex is much more soluble than is the complex with dithio-oxamide or thiourea, whilst the necessary time of heating is halved.
W. J. Baker

1379. **Spectrophotometric determination of rhodium with thiomalic [mercaptosuccinic] acid, and the simultaneous determination of rhodium and palladium.** V. L. Wagner, jun., and J. H. Yoe (Dept. of Chem., Univ. of Virginia, Charlottesville, U.S.A.). *Talanta*, 1959, **2** (3), 239–243.—From 2 to 7 p.p.m. of Rh in 25 ml of soln. can be determined by measuring the extinction at 340 mμ (1-cm quartz cells) of the yellow complex of Rh with 0·05 *M* mercaptosuccinic acid at pH 2. The colour is formed at 100° and the complex is stable for several days. The sensitivity is 0·055 p.p.m.; limiting permissible concn. of other ions are (for ≃4 p.p.m. of Rh)—Pt^{4+} 0·5, Pd^{2+} 0·05, Ru^{3+} 0·2, Os^{6+} 1·0, Ir^{4+} 1·5, and Au^{3+} 0·2 p.p.m. When Pd and Rh are both present (in ratios from 1 : 1 to 60 : 1) the extinctions can be measured on separate aliquots, at 350 mμ in each instance, but at 25° for Pd (*cf. Anal. Abstr.*, 1960, **7**, 1382) and 100° for Rh. The two extinctions are quant. additive; the accuracy is high both for Rh and Rh plus Pd.
W. J. Baker

1380. **Polarographic determination of rhodium.** J. Seifert and J. Šimek (Staatsanst. f. Edelmetalle, Prague). *Coll. Czech. Chem. Commun.*, 1959, **24** (6), 2046–2048 (in German).—The red modification of $RhCl_3$ can be determined polarographically with an accuracy of ±3%. All the platinum metals interfere but by the use of a separation technique the method can be applied to rhodium - platinum alloys containing ≃1% of Rh. Owing to the limitations of the method of separation the overall accuracy is ±10%. *Procedure*—Dissolve the sample (0·5 g) in aqua regia and evaporate to dryness. Add NaCl (2 to 3 g) and HCl (1 : 1) (10 ml). Evaporate to dryness and dilute to 200 ml with water. Add $NaHCO_3$ and $NaBrO_3$ (1 g) and digest on a water bath for 2 hr. Filter off the pptd. $Rh(OH)_3$ and dissolve it in the minimum amount of HCl (1 : 1). Evaporate to dryness, dissolve the residue in hot water, add gelatin soln. (0·5%) (1 ml) and sufficient HCl to give 25 ml of *M* soln. Measure the concn. of Rh polarographically against a mercury-pool electrode in the range −0·1 to −0·6 V.
W. T. Carter

1381. **Spectrophotometric determination of rhodium in uranium - rhodium alloys.** R. D. Gardner and A. D. Hues (Univ. of Calif., Los Alamos, N.M., U.S.A.). *Anal. Chem.*, 1959, **31** (9), 1488–1489.—Rhodium at concn. of about 1% can be determined rapidly without separation by measuring the extinction at 520 mμ of the red colour formed by the addition of $SnCl_2$ in HCl to the sample dissolved in HCl and H_2O_2. Only a few metals interfere at concn. equal to or less than that of the Rh. One-hundred determinations on 0·02 to 0·2 mg of Rh in the presence of 10 mg of U showed a relative standard deviation for individual values of ±0·8%.
K. A. Proctor

1382. **Spectrophotometric determination of palladium with thiomalic [mercaptosuccinic] acid.** V. L. Wagner, jun., and J. H. Yoe (Dept. of Chem., Univ. of Virginia, Charlottesville, U.S.A.). *Talanta*,

1959, **2** (3), 223–229.—From 0·4 to 10 p.p.m. of Pd in the sample soln. (25 ml) can be determined by measuring the extinction at 350 mμ (1-cm quartz cell) of the bright-yellow complex of Pd^{2+} with 0·01 *M* mercaptosuccinic acid formed at pH 1·1 to 2·6. The reaction is instantaneous and is independent of temp. in the range 15° to 35°; the complex is stable for several days. The sensitivity of the reaction is 0·05 p.p.m. of Pd; tolerances for other ions are listed. W. J. Baker

1383. **Spectrophotometric determination of osmium, platinum and ruthenium.** A. K. Majumdar and J. G. Sen Gupta (Jadavpur Univ., Calcutta). *Naturwissenschaften*, 1959, **46** (11), 374 (in English). —Anthranilic acid gives coloured complexes with Pt^{IV} and Ru^{III}, *m*-aminobenzoic acid with Os^{IV}, Os^{VI} and Os^{VIII}, 2-aminophenol-4-sulphonic acid with Pt^{IV}, Ru^{III}, Os^{VI} and Os^{VIII}, and 8-amino-1-naphthol-3:6-disulphonic acid with Os^{VI} and Os^{VIII}. The optimum pH for reaction, the absorption maxima and the concn. range over which Beer's law is obeyed are given in each case.
W. T. Carter

1384. **Quantitative determination of platinum in alumina-base reforming catalyst by X-ray spectroscopy.** A. J. Lincoln and E. N. Davis (Res. and Devel. Div., Engelhard Industries, Inc., Newark, N.J., U.S.A.). *Anal. Chem.*, 1959, **31** (8), 1317–1320.—The sample is prepared by grinding and calcining. Two methods of calibration are proposed, depending on the use of a tungsten or molybdenum target X-ray tube. The standard deviation is $\simeq$ $\pm$0·0025 for 0·6% of Pt, and the accuracy compares well with that of spectrophotometric methods. A sample can be analysed in about 3 hr., about 30 min. of which is operator time. K. A. Proctor

1385. **Neutron activation analysis of rocks and ore concentrates.** D. I. Leĭpunskaya, Z. E. Gauer and G. N. Flerov. *Atomnaya Energiya*, 1959, **6** (3), 315–319.—Portable polonium - beryllium sources (1·5 to 2 C and intensity 2×10^7 neutrons per sec.) for use in the field for mineral prospecting were investigated. A very rapid method for use with both fast and slow neutrons is described for the determination of Al and Si in bauxite, Mn, Fe and V in iron ore, and In in polymetallic ores and concentrates; a short-lived isotope of the element being determined is used as an indicator. Induced β-activity is measured by means of an ordinary counter placed in the centre of a double-walled cylindrical polyethylene container filled with the material to be exposed; γ-activity is measured by means of a scintillation γ-spectrometer with a one-channel amplitude analyser. The samples are exposed for 5 to 20 min. and the measurement of induced activity takes 5 to 10 min. The method is highly sensitive and far more accurate than photometric and spectrographic methods. K. R. C.

1386. **Fluorescence X-ray spectrometric estimation of aluminium, silicon and iron in the flotation products of clays and bauxites.** Shiou-Chuan Sun (Coll. of Mineral Industries, Pennsylvania State Univ., University Park, U.S.A.). *Anal. Chem.*, 1959, **31** (8), 1322–1324.—Aluminium and Si are determined with ethylenediamine tartrate as the analysing crystals in a helium atmosphere, and Fe with LiF as the analysing crystals in air. The mean error, compared with chemical results, is $\simeq$5% (max. 11%). The lower limit of detection of each element is $\simeq$0·2%. A complete analysis of one sample takes $\simeq$2 hr., but on a routine basis <1 hr. is required.
K. A. Proctor

1387. **Spectrographic analysis of refractory clays and fireclay refractories.** A. F. Kolechkova. *Fiz. Sb. L'vovsk. Univ.*, 1958, [4(9)], 474–477; *Ref. Zhur., Khim.*, 1959, (18), Abstr. No. 64,157.—The samples are ignited, sieved (10,000 meshes per sq. cm), mixed with carbon powder in the ratio 1:3, and placed in a cavity in a carbon electrode; the external diam. of the upper portion of the electrode is 4 mm, and the cavity has a diam. of 2 mm and a depth of 3 mm; the upper electrode is sharpened to a truncated cone; 10% of CoO is added to samples and standards. The spectra are excited in an a.c. arc at 10 amp., with an arc gap of 2·5 mm and a secondary spark gap of 0·5 mm. The medium spectrograph slit of width 0·02 mm is illuminated for 40 sec. without preliminary ignition. Calibration curves of $\triangle S$ *vs.* log *C* are constructed from the lines (in Å) Ti 3191·9 and Co 3188·3, Fe 3047·6 and Co 3050·9, Ca 3179·3 and Co 3177·2, and Mg 2779·8 and Co 2764·1. The reproducibility is within 4·5%. The discrepancy between the results of the chemical and spectrographic analyses is within the limits required by GOST 2642–53. Standards are prepared on a kaolin base with the addition of the oxides. The error in determining Si and Al is considerably higher than the error of chemical analysis; the spectrographic determination of Ti, Fe, Ca and Mg is therefore combined with a rapid chemical analysis for Si and Al.
C. D. Kopkin

1388. **Analysis of refractory borides, carbides, nitrides and silicides.** O. H. Kriege (Los Alamos Sci. Lab., N. Mex.). *U.S. Atomic Energy Comm.*, Rep. LA-2306, 1959, 58 pp.—Methods are presented for the analysis of 41 refractory materials. An evaluation of the accuracy and the precision of these techniques is also given. The materials studied are the borides of Hf, Mo, Nb, Re, Ta, Th, Ti, W, U, V and Zr, the carbides of Hf, Mo, Nb, Si, Ta, Th, Ti, W, U, V and Zr, the nitrides of B, Hf, Nb, Si, Ta, Ti, U and Zr, the silicides of Mo, Re, Ta, Ti, W, V and Zr, and the mixed carbides of U with Hf, Nb, Ta or Zr. Nucl. Sci. Abstr.

1389. **Chemical analysis of silica bricks for the arched roofs of open-hearth furnaces.** P. Thiery and J. Baron. *Chim. et Ind.*, 1959, **81** (5), 681–689.—A scheme of analysis is described, based on American, French, German and British standard procedures. For the determination of SiO_2 only, attack with HF - HNO_3 has the advantage of bringing various constituents into the form of nitrates (which are then converted into oxides) and not of sulphates, which necessitates a subsequent determination of SO_3 owing to their incomplete dissociation during calcination. For determining the other constituents, the bricks are dissolved in HF - H_2SO_4 and it is essential that the amount of H_2SO_4 should be sufficient to dissolve the fluorides in order to prevent loss due to volatilisation of these compounds and the possible presence of residual fluorine which would interfere with the pptn. of the hydroxides. Discrepancies between the results obtained by different laboratories for the Al_2O_3 content determined by difference were found to be due to the presence of SiO_2 in the oxides R_2O_3, and sometimes to a loss of wt. by the platinum crucible.
J. M. Jacobs

1390. **Spectrographic analysis of acid tin- and nickel-plating baths.** T. V. Bȳkova and B. M. Yakovlev. *Fiz. Sb. L'vovsk. Univ.*, 1958, [4(9)], 510–512; *Ref. Zhur., Khim.*, 1959, (18), Abstr. No. 64,180.—Place 1 drop of a soln. of the electrolyte on a copper electrode sharpened to a truncated cone of diam. 4 mm near the top, and evaporate the dry residue in an a.c. arc at 2·5 amp. with an arc gap of 1·5 mm. In the analysis of acid tin-plating baths, add Ni to the soln. of samples and standards; in the determination of Ni in nickel-plating baths add Mn, and in the determination of B add Mo. The analysis is carried out from calibration curves of $\triangle S$ *vs.* log C and the lines (in A) Sn 2706·5 and Ni 3050·8, Na 3302·9 and Ni 3050·8, Ni 3050·8 and Mn 2576·1, and B 2496·7 and Mo 2775·4. The error is $\not> 5\%$. C. D. Kopkin

1391. **Spectrographic analysis of Alundum and glass.** A. N. Prokop'eva. *Fiz. Sb. L'vovsk. Univ.*, 1958, [4(9)], 446–450; *Ref. Zhur., Khim.*, 1959, (18), Abstr. No. 64,174.—The sample of glass, ground to 200 mesh, is mixed with carbon powder in the ratio 1:2 and the mixture is placed in a cavity in a copper electrode. The spectra are excited in an a.c. arc at 5 amp. with an upper copper electrode. In the determination of Pb, K, Al, Ca, Na, Mg and Fe, the background is used as internal standard. The slit of a medium quartz spectrograph is illuminated for 10 sec. The determination of B is carried out in a h.f. spark, the sample of glass being mixed with nickel powder in the ratio 1:10; a pellet is made from the mixture, and is used as the lower electrode. Chemically analysed samples of glass are used as standards; the type of glass used must be the same as the sample. Crude Alundum powder is mixed with carbon in the ratio 1:1 and the mixture is spread in a thin layer on a copper plate, moving at a rate of 16 cm per minute, which serves as lower electrode. The slit is illuminated for 30 sec. Alundum coatings taken from fabricated parts are fixed to the moving copper electrode with a thin layer of Bakelite varnish. Calibration curves are constructed of $\triangle S$ *vs.* log C. C. D. Kopkin

1392. **Examination of fuel ash. Chelatometric determination of iron and aluminium.** W. Radmacher and W. Schmitz (Brennstoffchem. Inst., Ruhrkohlen-Beratung G.m.b.H., Essen, W. Germany). *BrennstChemie*, 1959, **40** (5), 161–164.—The ash is decomposed with a mixture of $KNaCO_3$ and $Na_2B_4O_7$. If only Fe and Al are to be determined, SiO_2 need not be removed, since silicate ions do not interfere with the titrations. Iron is determined by titrating an aliquot of the soln., adjusted to pH 1·8, with 0·05 M EDTA (disodium salt), with salicylic acid as indicator. After titration, the colourless or yellow soln. is adjusted to pH 3 with acetic acid and ammonium acetate, heated to boiling-point while aq. Cu - EDTA and ethanolic 1-(2-pyridylazo)-2-naphthol are added, and titrated with 0·05 M EDTA until the red colour no longer returns after boiling for 1 min. (*cf.* Flaschka and Abdine, *Anal. Abstr.*, 1957, **4**, 414). Excluding the removal of SiO_2, the analysis takes 30 min. Results agree with those by titration with Ti^{3+} and pptn. as phosphates, and by photometry. A. R. Pearson

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W. J. BAKER

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E. J. H. BIRCH

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C. A. FINCH

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L. ZANONI

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D. F. PHILLIPS

1404. **Analysis of organic compounds by flame spectrometry. I. Determination of chlorine in organic compounds by band spectra of cuprous chloride.** Masao Maruyama and Setsuya Seno (Shinagawa Plant, Sankyo Co. Ltd., Nishi-Shinagawa, Shinagawa-ku, Tokyo). *Bull. Chem. Soc. Japan*, 1959, **32** (5), 486–490 (in English).—This determination is carried out by using dimethylformamide containing Cu as a solvent [12·08 g of $Cu(NO_3)_2.3H_2O$ is dissolved in dimethylformamide, 1 ml of HNO_3 is added, and the mixture is made up to 1 litre] and a peak of the band spectrum of Cu_2Cl_2 at 435·4 mμ. To decrease the effect of the C_2-Swan band that is produced on the combustion of organic compounds, and to increase the sensitivity of the Cu_2Cl_2 band spectrum, the combustion gas is maintained at a high pressure of O (25 p.s.i.) and a low pressure of H (1st, 0·5 p.s.i.) (2nd, 10 cm water column). Good results (≃98 to 102% of theoretical

1390. **Spectrographic analysis of acid tin- and nickel-plating baths.** T. V. Bÿkova and B. M. Yakovlev. *Fiz. Sb. L'vovsk. Univ.*, 1958, [4(9)], 510–512; *Ref. Zhur., Khim.*, 1959, (18), Abstr. No. 64,180.—Place 1 drop of a soln. of the electrolyte on a copper electrode sharpened to a truncated cone of diam. 4 mm near the top, and evaporate the dry residue in an a.c. arc at 2·5 amp. with an arc gap of 1·5 mm. In the analysis of acid tin-plating baths, add Ni to the soln. of samples and standards; in the determination of Ni in nickel-plating baths add Mn, and in the determination of B add Mo. The analysis is carried out from calibration curves of $\triangle S$ *vs.* log C and the lines (in Å) Sn 2706·5 and Ni 3050·8, Na 3302·9 and Ni 3050·8, Ni 3050·8 and Mn 2576·1, and B 2496·7 and Mo 2775·4. The error is $\not> 5\%$. C. D. Kopkin

1391. **Spectrographic analysis of Alundum and glass.** A. N. Prokop'eva. *Fiz. Sb. L'vovsk. Univ.*, 1958, [4(9)], 446–450; *Ref. Zhur., Khim.*, 1959, (18), Abstr. No. 64,174.—The sample of glass, ground to 200 mesh, is mixed with carbon powder in the ratio 1:2 and the mixture is placed in a cavity in a copper electrode. The spectra are excited in an a.c. arc at 5 amp. with an upper copper electrode. In the determination of Pb, K, Al, Ca, Na, Mg and Fe, the background is used as internal standard. The slit of a medium quartz spectrograph is illuminated for 10 sec. The determination of B is carried out in a h.f. spark, the sample of glass being mixed with nickel powder in the ratio 1:10; a pellet is made from the mixture, and is used as the lower electrode. Chemically analysed samples of glass are used as standards; the type of glass used must be the same as the sample. Crude Alundum powder is mixed with carbon in the ratio 1:1 and the mixture is spread in a thin layer on a copper plate, moving at a rate of 16 cm per minute, which serves as lower electrode. The slit is illuminated for 30 sec. Alundum coatings taken from fabricated parts are fixed to the moving copper electrode with a thin layer of Bakelite varnish. Calibration curves are constructed of $\triangle S$ *vs.* log C. C. D. Kopkin

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values) are obtained with samples other than liquids, metal salts and hydrochlorides. The determinable range for Cl is 0·005 to 0·1 *M*. I. JONES

1405. **Volumetric determinations in strongly alkaline media. X. The determination of active methylene groups.** O. Ryba (Research Inst. for Photograph. Chem., Prague). *Coll. Czech. Chem. Commun.*, 1959, **24** (6), 1950–1953 (in German).—*p*-Amino-NN-diethylaniline is oxidised by $K_3Fe(CN)_6$ in the alkaline soln. to give a quinonedi-imine which reacts with active methylene groups to give leuco bases, and these are oxidised by $K_3Fe(CN)_6$ to give the corresponding dyes. This series of reactions is utilised for the potentiometric titration of compounds containing active methylene groups, with special reference to the substances used in the manufacture of colour film. *Procedure*—To 10 to 20 ml of a soln. of the sample add *p*-amino-NN-diethylaniline soln. (0·05 *M*) (5 ml) and K_2CO_3 soln. (0·1 *M*) (20 ml). Dilute to 50 ml with water and titrate potentiometrically with $K_3Fe(CN)_6$ soln. (0·1 *N*), with a platinum - S.C.E. pair. Carry out a blank titration on the reagents [1 ml of 0·1 *N* $K_3Fe(CN)_6 \equiv 0{\cdot}701$ mg of $-CH_2-$]. The end-point can be determined to within $\pm 0{\cdot}03$ ml. W. T. CARTER

1406. **New method of hydrocarbon structural group analysis.** D. S. Montgomery and M. L. Boyd (Dept. of Mines and Tech. Surveys, Ottawa, Canada). *Anal. Chem.*, 1959, **31** (8), 1290–1298.—Three chemical and two physical properties are expressed in terms of five structural groups as equations that can be simultaneously solved by computers. The chemical properties are the carbon and hydrogen content and the number of aromatic C atoms per molecule. The physical properties required for the analysis are the molecular volume and the molar refraction. The method was applied to 114 hydrocarbons whose properties had been tested by A.P.I. Project 42. G. P. COOK

1407. **Infra-red absorption spectra in the caesium bromide region (15 to 35 microns). I. Hydrocarbons.** A. Cornu. *Bull. Soc. Chim. France*, 1959, (5), 721–729.—The i.r. spectra of 96 hydrocarbons in this region are mapped. E. J. H. BIRCH

1408. **Polarographic determination of hexaethyldilead in tetraethyl-lead.** L. N. Vertyulina and I. A. Korshunov. *Khim. Nauka i Prom.*, 1959, **4** (1), 136; *Ref. Zhur., Khim.*, 1959, (18), Abstr. No. 64,250.—A method is proposed for the determination of hexaethyldilead (**I**) in admixture with tetraethyl-lead and triethyl-lead chloride. Because of the ease of hydrolysis of **I**, the determination is carried out in anhyd. ethanol, with tetraethylammonium iodide as basal electrolyte. The $E_{\frac{1}{2}}$ of **I** is 1·8 to 2·0 V. The determinable limits of concn. of **I** are 0·5 to 10·0%; the mean error is 7%. The sample soln. must be free from O. C. D. KOPKIN

1409. **An investigation into the action of bases on chloroform.** H. Williams (Welsh Sch. of Pharm., Coll. of Adv. Technol., Cardiff). *J. Pharm. Pharmacol.*, 1959, **11** (7), 400–410.—The presence of reactive halogeno-hydrocarbons as impurities in chloroform B.P. has been confirmed (*cf.* Caws and Foster, *Anal. Abstr.*, 1958, **5**, 2760). It is shown that strong bases (*e.g.*, piperidine and pyrrolidine) react with these impurities, and can be used to purify $CHCl_3$. To assess the quality of a sample of chloroform, heat the sample (3·7 g) with piperidine (0·85 g) in a sealed ampoule at 60° for 2 hr., neutralise with 6 *N* HNO_3, add a further portion of HNO_3 (1 ml) and an excess of 0·02 *N* $AgNO_3$ and back-titrate with 0·02 *N* NH_4SCN in the presence of nitrobenzene (1 ml), with $(NH_4)_2SO_4.Fe_2(SO_4)_3$ as indicator. The halide ion liberated from pure $CHCl_3$ consumes <1 ml of 0·02 *N* $AgNO_3$; a typical sample of chloroform B.P. requires 8 ml. A. R. ROGERS

1410. **Determination of higher primary and secondary alcohols by the dehydration method.** A. N. Bashkirov, S. A. Lodzik and V. V. Kamzolkin. *Trudy Inst. Nefti, Akad. Nauk SSSR*, 1958, **12**, 297–298; *Ref. Zhur., Khim.*, 1959, (16), Abstr. No. 56,939.—Dehydrate 3·5 g of an alcohol (C_8 to C_{18}) by boiling with 50 ml of xylene for 3 hr. (at 140° to 142°) in the presence of 3·5 g of microspherical aluminosilicate catalyst in a Dean and Stark apparatus, and measure the vol. of water liberated. Primary alcohols do not react, but secondary alcohols are quantitatively dehydrated. The method may be used for the analysis of mixtures of primary and secondary alcohols obtained by the direct oxidation of paraffins. The max. error is 4·5%. C. D. KOPKIN

1411. **Paper ionophoresis of carbohydrates. I. Procedures and results for four electrolytes.** J. L. Frahn and J. A. Mills. *Aust. J. Chem.*, 1959, **12**, 65–89.—Full analytical details are given of the procedures for paper ionophoresis of polyhydric compounds in the electrolytes sodium borate, sodium arsenite, basic Pb acetate and NaOH, and for detecting the compounds after ionophoresis. Some new or improved reagents have been developed for locating the spots on paper strips under various conditions. Rates of migration for 96 compounds, including many pentoses and hexoses, the common disaccharides, all sugar alcohols up to the heptitols, the cyclitols, a number of glycols, several glycosides and other carbohydrate derivatives are given. Sodium arsenite and basic Pb acetate are the most effective electrolytes for separating reducing sugars, basic Pb acetate is the best for separating sugar alcohols, and sodium borate is the best for simple glycols. In particular, mannose and rhamnose migrate slowly in sodium borate but fairly rapidly in basic Pb acetate; xylose, glucose, arabinose and galactose migrate very rapidly in sodium borate, but very slowly in basic Pb acetate; galactose and glucose may be separated more easily in sodium arsenite than in sodium borate; fructose and sorbose have characteristically high mobilities in sodium arsenite. The correlation of the configuration of stereoisomers with their mobilities is discussed.

SUGAR IND. ABSTR.

1412. **Paper chromatography of sugar phenylosazones as their borate complexes.** B. Arreguín (Ciudad Univ., Mexico, D.F.). *Anal. Chem.*, 1959, **31** (8), 1371–1372.—The phenylosazones were applied to S. & S. No. 2040 paper that had been impregnated with 0·1 *M* $K_2B_4O_7$ and dried at 60°. The solvent systems that gave the best results were, (*i*) 75% aq. dioxan soln. - hexane - H_2O (2:2:1), (*ii*) 75% aq. dioxan soln. - hexane - H_2O - benzene (11:10:5:4), and (*iii*) 75% aq. dioxan soln. - H_2O - 1:2-dichloroethane (27:20:3). The spots are yellow or faintly orange and are readily observed through a blue or violet filter; 15 to 25 μg can be detected. R_F values for 12 compounds in each of the three solvent systems are listed. G. P. COOK

1413. Stoicheiometry of chlorite - aldehyde reactions. Analytical procedures. H. F. Launer and Yoshio Tomimatsu (West Reg. Res. Lab., Albany, Calif., U.S.A.). *Anal. Chem.*, 1959, **31** (8), 1385–1390.—Sodium chlorite shows no fixed stoicheiometric relationship to aldehyde groups in reactions with aldoses, benzaldehyde and dextran dialdehyde. The ratios varied with the aldehyde, the kind and concn. of buffer soln. used, the reaction rate and other factors. Ratios of 2·63 to 3·77 were obtained with 0·5 *M* phosphate buffer which limits its usefulness to known aldehydes. Ratios of 2·56 to 2·79 were given with 3 *M* phosphate buffer and permitted the use of a mean value in the analysis of unknown aldehydes, including polysaccharides. G. P. Cook

1414. Spectrophotometric determination of acetylacetone with cupric acetate. R. J. Starkey (Courtaulds Ltd., Coventry, England). *Analyst*, 1959, **84**, 517–519.—The sample is shaken with cupric acetate soln. and a Na acetate buffer soln., and the mixture is extracted with CCl_4 that has been saturated with the Cu acetylacetone chelate derivative; the extract is discarded. The pptd. blue complex in the aq. layer is extracted with $CHCl_3$, and the extinction of the extract is measured at 650 mμ. The precision is within $\pm 1\%$ and the accuracy within $\pm 1{\cdot}5\%$, even with discoloured multi-component mixtures. A. O. Jones

1415. Determination of *vic.*-dioximes. C. V. Banks and J. J. Richard (Iowa State Coll., Ames, U.S.A.). *Talanta*, 1959, **2** (3), 235–238.—The purity of an aliphatic or alicyclic *vic.*-dioxime can be determined by quant. oxidation of the *vic.*-dioxime group to the furoxan structure, mol. for mol. *Procedure*—Dissolve the sample (containing 2 to 7×10^{-4} mole of *vic.*-dioxime) in CCl_4 (100 ml), add 0·1 *N* iodine (25 ml) plus 3% (w/v) soln. of mercuric acetate in acetic acid (25 ml), followed, after $\simeq 30$ min., by KI soln. (7·5%, w/v) (75 ml). Shake the mixture vigorously and then titrate the excess of iodine with 0·1 *N* $Na_2S_2O_3$ to the usual starch end-point. Results for 15 different compounds (listed) are satisfactory; benzil α-dioxime, diaminoglyoxime and furil α-dioxime are not amenable to this method. The chief interferences in the titration are easily oxidised or halogenated compounds, primary amines, ketoximes, aldoximes and monoximes of *vic.*-diketones. W. J. Baker

1416. Application of the electrometric contact method to the paper chromatography of some organic acids. B. Zapiór and J. Piatek (Dept. of Gen. Chem., Jagellonian Univ., Kraków). *Roczn. Chem.*, 1959, **33** (4–5), 1159–1165.—The indicating antimony micro-electrode of Kamieński *et al.* (*Bull. Acad. Polon. Sci., III*, 1953, **1**, 1) has been applied to the detection of tartaric, citric and succinic acids on paper chromatograms, developed with *n*-butanol - 85% formic acid - water (12:1:1) (Barnabas and Joshi, *Anal. Chem.*, 1955, **27**, 443). After drying the paper at room temp. or in a current of warm air, traces of formic acid left were neutralised by spraying with 0·01% aq. KOH soln. The chromatogram was then laid on a glass plate provided with a millimetre scale and wetted with 0·1 *N* KCl, and the electrode was applied at 2·5 to 5-mm intervals, and cleaned with filter-paper wetted with 0·1 *N* KCl after each contact with the paper. From graphs of mV *vs.* distance from the origin, the following R_F values were obtained for samples of 50 μg and 5 μg, respectively—tartaric acid, 0·26, 0·24; citric acid, 0·44, 0·43; succinic acid, 0·74 and 0·73. These values were confirmed by conventional spray methods of detection. The min. amount of acid giving a stable potential change was 5 μg, and this quantity of one acid could be detected in the presence of 50 μg of the others. W. Roubo

1417. Identification of the C_6 to C_{11} fatty acids by paper chromatography. M. Romaňuk (Czech. Acad. Sci., Prague). *Coll. Czech. Chem. Commun.*, 1959, **24** (6), 2064–2065 (in German).—R_F values are given for the separation of the acids by descending chromatography on No. 1 Whatman paper, with 2-ethylhexanol satd. with 5 *N* aq. NH_3 as mobile phase. Development is carried out for 18 hr., and after drying in air for 7 hr. the separated substances are detected by spraying with chlorophenol red. W. T. Carter

1418. Determination of $\alpha\beta$-unsaturated acids and esters by bromination. F. E. Critchfield (Union Carbide Chemicals Co., S. Charleston, W. Va., U.S.A.). *Anal. Chem.*, 1959, **31** (8), 1406–1408.—Under ordinary conditions $\alpha\beta$-unsaturated acids and esters react slowly with Br but, with the corresponding sodium or potassium salt, bromination proceeds rapidly and smoothly. The acids are neutralised with NaOH and the esters are saponified with KOH before bromination. The method was applied to the assay of 14 acids and esters, including maleic and fumaric esters which are difficult to analyse by other methods. The precision is $\pm 0{\cdot}1\%$, and the results agreed to within 0·7% with those obtained by other methods. G. P. Cook

1419. Conductimetric titration of carboxylic and phenolic acids in non-aqueous solutions. II. Titration of dibasic acids. N. van Meurs and E. A. M. F. Dahmen [Koninklijke/Shell Lab. (N.V. De Bataafsche Petroleum Maatschappij, Amsterdam, Netherlands]. *Anal. Chim. Acta*, 1959, **21** (1), 10–16 (in English).—The conductimetric titration of dibasic acids with tetramethylammonium hydroxide as a base sometimes gives rise to N-shaped titration curves and sometimes to chair-shaped curves. The factors determining the shape of the curves are discussed, *viz*, the distance between the acid groups, the solvating power and dielectric constant of the solvent, the size of the base cation and the temp. of titration. The optimum conditions for titration are—the use of solvents of low solvating power (*e.g.*, pyridine or dimethylformamide), and titration at high temp. (up to 70°). T. R. Andrew

1420. Determination of inorganic sulphate in sodium lauryl sulphate. J. R. Gwilt and J. S. Hedley (Winthrop Laboratories Ltd., Newcastle upon Tyne, England). *J. Pharm. Pharmacol.*, 1959, **11** (7), 442–444.—Dissolve the sample (1 g) in warm H_2O (35 ml), add 5% HNO_3 (2 ml) and ethanol (50 ml) and heat to boiling. Slowly add 0·1 *M* $Pb(NO_3)_2$ (10 ml) with stirring and simmer for 5 min. Allow the ppt. to settle, decant the supernatant liquid through filter-paper and wash the ppt. by decantation with boiling 50% ethanol ($4 \times$ 50 ml). Transfer the filter-paper to the vessel which contains the bulk of the ppt. and immediately add H_2O (40 ml), 0·1 *M* EDTA (disodium salt) (10 ml) and buffer soln. [dissolve NH_4Cl (8·25 g) in H_2O, add aq. NH_3 (*d*, 0·880) (113 ml) and dilute to 1 litre] (5 ml). Warm to dissolve the ppt. and titrate with 0·05 *M* $ZnSO_4$, with Eriochrome black T as indicator. The method is more precise than that of the B.P., and a determination can be completed in 30 to 40 min. A. R. Rogers

1421. **Determination of gluconolactone, galactonolactone and their free acids by the hydroxamate method.** O. G. Lien, jun. (Sch. of Public Health, Univ. of Calif., Berkeley, U.S.A.). *Anal. Chem.*, 1959, **31** (8), 1363–1366.—The free acids are converted into their lactones by acidification with HCl to between pH 1·5 and 2·0 followed by heating at 120° in an autoclave at 15 p.s.i. The samples are then cooled, hydroxylamine soln. (at pH 8), HCl and $FeCl_3$ soln. are added and the extinction is measured at 540 mμ. With lactone samples the initial acidification and heating steps are omitted.
G. P. Cook

1422. **Mass-spectrometric studies. I. Methyl esters of saturated normal-chain carboxylic acids.** R. Ryhage and E. Stenhagen (Kem. Inst. I, Karolinska Inst., Stockholm). *Ark. Kemi*, 1959, **13** (6), 523–542 (in English).—Details of the mass spectra are given for the methyl esters of saturated normal-chain carboxylic acids up to and including methyl *n*-hexacosanoate (*cf.* Hallgren *et al.*, *Anal. Abstr.*, 1958, **5**, 1698). N. E.

1423. **Determination of water in recirculated butyl acetate.** Z. Łada (Inst. of Gen. Chem., Warsaw). *Rev. Chim., Bucharest*, 1959, **10** (6), 348–349.—The Fischer method is shown to be adequate, and uninfluenced by the presence of other normal impurities, the error being $\not> 0·11\%$ on a water content of 3·6%. H. Sher

1424. **Chromatographic separation of C_1 to C_6 primary and C_2 to C_{12} secondary monoamines as 3:5-dinitrobenzamides.** D. P. Johnson and J. B. Johnson (Union Carbide Chemicals Co., S. Charleston, W. Va., U.S.A.). *Anal. Chem.*, 1959, **31** (8), 1373–1374.—The 3:5-dinitrobenzamides are separated on a silicic acid column with *n*-hexane - diethyl ether mixtures as the developing solvents. The zones are located by treating eluate fractions with acetone and aq. KOH soln. to form the quinonoid ion colour. R_F values are listed for all primary and secondary amines from methyl to hexyl. G. P. Cook

1425. **Volumetric estimation of thiourea in presence of mercuric chloride.** Satyendra Nath Banerjee (Chem. Technol. Dept., Univ. of Bombay). *J. Indian Chem. Soc.*, 1959, **36** (6), 449–450.—The determination of thiourea (**I**) in the presence of varied amounts of $HgCl_2$ (**II**) with $KBrO_3$ - KBr soln. as titrant at 35° in the presence of H_2SO_4 and KI, with starch as indicator (Mahr, *Z. anal. Chem.*, 1939, **117**, 91), is studied. For 10 ml of 0·2 *N* **I** and 1 ml of 4% **II** soln., 10 to 15 ml each of 18 *N* H_2SO_4 and 1% KI soln. are required. Greater or less amounts of acid lead to high results. In the presence of greater amounts of **II**, sufficient KI soln. must be added to keep the Hg in solution as HgI_4^{2-}.
I. Jones

1426. **Potentiometric and heterometric titration of thioacetamide solutions with silver nitrate.** E. Bovalini and M. Piazzi (Univ. Siena, Italy). *Ann. Chim., Roma*, 1959, **49** (5–6), 1067–1074.—Potentiometric and heterometric titrations of thioacetamide soln. (0·1 to 0·001 *M*) are studied with respect to the conditions of pH and concn. Concordant and reproducible results can be obtained, permitting a good standardisation of thioacetamide soln. and an accurate heterometric micro-determination of Ag. Standard procedures and suitable curves are given for both methods.
C. A. Finch

1427. **Gas - liquid partition chromatography of sulphur compounds with ββ′-iminodipropionitrile.** J. H. Karchmer (Humble Oil and Refining Co., Baytown, Tex., U.S.A.). *Anal. Chem.*, 1959, **31** (8), 1377–1379.—The behaviour of 11 compounds in the 58° to 126° boiling-range has been investigated on a column of 22% of ββ′-iminodipropionitrile on Celite at 84° with a helium flow-rate of 70 ml per min. The identification of various types of sulphur compound, such as thiols and thiophens, is possible with a dual stationary-phase technique.
K. A. Proctor

1428. **The near u.v. spectra of thionylamines.** G. Leandri and A. Mangini (Ist. Chim. Ind., Univ., Bologna, Italy). *Spectrochim. Acta*, 1959, (6), 421–431.—Twenty-six thionylamines (R·NSO) (18 with R an aromatic radical) have been examined between 200 and 370 mμ in *cyclo*hexane or *n*-hexane soln. Two regions of absorption are noted—R aliphatic, λ_{max} 235 to 239 mμ (log ε 3·63 to 3·71) and λ_{max} 300 to 312 mμ (log ε 1·51 to 1·69); R aromatic, λ_{max} 232 to 270 mμ (log ε 3·73 to 4·11) and λ_{max} 308 to 408 mμ (log ε 3·21 to 4·45). The interpretation of the spectra and the effects of substitution are discussed. E. G. Cummins

1429. **High-temperature gas chromatography of aromatic hydrocarbons: instrument design and exploratory studies at temperatures up to 430°.** R. A. Baxter and R. T. Keen (Atomics International Div., N. Amer. Aviation Inc., Canoga Park, Calif.). *U.S. Atomic Energy Comm.*, Rep. NAA-SR-3154, 1959, 30 pp.—A gas chromatograph capable of successful operation at temp. up to 430° has been constructed and evaluated. The important features and operating characteristics of the instrument are described. Several stationary phases have been tested for partitioning quality and high-temp. stability. The best material was a polyphenol-type tar with an average mol. wt. of 2100. The high-temp. technique has been used for both quant. and qual. analysis of irradiated polyphenol reactor coolant-moderator materials. At 430° it has provided the means for analysing mixtures of compounds having five or six aromatic rings.
Nucl. Sci. Abstr.

1430. **Colour reactions of certain phenols with Ehrlich's reagent.** C. Steelink (Dept. Chem., Univ. Arizona, Tucson, U.S.A.). *Nature*, 1959, **184** (Suppl. No. 10), 720.—Chromatograms were developed in 5% (v/v) aq. acid at 24° for 3·5 hr. They were air-dried and then sprayed with either Ehrlich's reagent (1% *p*-dimethylaminobenzaldehyde in *N* HCl) or cinnamaldehyde - HCl reagent (*cf.* Block *et al.*, "Manual of Paper Chromatography and Paper Electrophoresis," 2nd. Ed., Academic Press Inc., N.Y., 1958). It was found that phloroglucinol-derived compounds reacted immediately with Ehrlich's reagent, and resorcinol-derived compounds only after heating. A list of compounds with the corresponding R_F values and their characteristic colours with both reagents is given.
S. Baar

1431. **Bromimetric determination of phenol and cresol isomers.** J. Młodecka (Inst. of Gen. Chem., Warsaw). *Rev. Chim., Bucharest*, 1959, **10** (6), 343–347.—The method establishes conditions for bromination such that errors are reduced to <2% for phenol alone and <3% for mixtures on a total quantity of 5 to 50 mg. *Procedure*—The soln. (20 ml) containing 0·005 to 0·05 g of cresol mixture

is treated with 5 ml of conc. HCl. From a burette 0·1 N bromate - bromide soln. is run in until a yellow colour appears; a further 20 to 25% of the vol. already used is then added and the titration flask is closed. After exactly 2 min., 2 g of KI is added and the mixture is set aside for 5 min. and then titrated with 0·02 N $Na_2S_2O_3$, with starch as indicator. H. SHER

1432. **Pentachlorophenol.** British Standards Institution (2 Park St., London, W.1). B.S. 3175: 1959, 8 pp.—Methods are described for the determination of crystallising-point, matter insol. in NaOH soln., and total acidity.

1433. **Separation and analysis of dihydric phenols by gas chromatography.** J. Janák and R. Komers (Czech. Acad. Sci., Brno). *Coll. Czech. Chem. Commun.*, 1959, **24** (6), 1960–1966 (in German).—Elution volumes are given for catechol and its homologues when galactonic acid lactone, dulcitol, inositol, mannitol and dimethylpolysiloxane are used as stationary phases. The relationship between molecular structure and elution volume is discussed and the use of the method for the qual. and quant. analysis of dihydric phenols in lignite coal-tar fractions is described. W. T. CARTER

1434. **Ultra-violet spectrophotometric determination of phthalic acid. Application to analysis of naphthalene oxidation products.** W. P. Fassinger and C. E. Gonter (Res. and Devel. Dept., Pittsburgh Coke and Chem. Co., Pa., U.S.A.). *Anal. Chem.*, 1959, **31** (8), 1324–1326.—Phthalic acid is measured at 274 mμ in 0·1 M HCl soln. with little or no interference from maleic acid. Both acids are pptd. as their barium salts from ethanol to separate them from 1:4-naphthaquinone and other compounds that interfere at this wavelength. The precision and accuracy are within $\pm$1·5% for concn. of <4 g per litre, and were calculated from duplicate determinations of five samples. Results compared well with those obtained by a polarographic titration method. G. P. COOK

1435. **Spectrophotometric determination of apiole and analogous compounds containing methylenedioxy groups by reaction with gallic acid and chromotropic acid.** E. Bovalini and A. Casini (Univ. Siena, Italy). *Ann. Chim., Roma*, 1959, **49** (5–6), 1059–1066.—The reactions of apiole, piperonal and safrole with gallic acid (the Labat reaction) and chromotropic acid are examined. The absorption spectrum of the colour obtained with gallic acid is recorded, and the relation between concn. and absorption for the two reactions is studied. The spectra are compared with those obtained with formaldehyde and trioxan, and a quant. photometric determination of these compounds is suggested. With the Labat reaction an accuracy within 0·5% is attained, and with chromotropic acid the accuracy appears to be within 0·3%. C. A. FINCH

1436. **Potentiometric titration of aromatic diamines with nitrite.** M. Matrka and Z. Ságner (Res. Inst. Org. Synth., Pardubice-Rybitví, Czechoslovakia). *Chem. Průmysl*, 1959, **9** (6), 288–291.—The usual method for the titration of primary aromatic amines with nitrite is also suitable for the titration of *o*- and *p*-phenylenediamine and benzidine in a HCl medium, with an accuracy of $\pm$0·5%. The mechanism of the reaction is discussed. *Procedure*—Dissolve the sample ($\equiv$0·001 mole) in N HCl (100 ml) and titrate potentiometrically with 0·2 N $NaNO_2$. The quant. course of the titration of *m*-phenylenediamine depends on the concn. of HCl, so that unreliable results were obtained with this isomer. J. ZÝKA

1437. **Detection and spectrophotometric determination of the phenyl ester of methylcarbamic acid.** E. Maggiorelli (Ist. Chim. Farm. Milit., Firenze, Italy). *Farmaco, Ed. Prat.*, 1959, **14** (5), 289–294.—Five reactions for the detection of methylcarbamic acid are given and the following method of determination is proposed. The substance (200 to 400 μg) is dissolved in 3 ml of glacial acetic acid, 1 ml of conc. HNO_3 and 1 ml of conc. H_2SO_4 are added and the soln. is heated on a boiling-water bath for 1 hr. After cooling, the liquid is transferred to a 100-ml flask containing 25 ml of 7 N NaOH and made up to volume with water, and the extinction is measured at 386 mμ. L. ZANONI

1438. **Gas - liquid partition chromatography of isomeric alkylcyclopentenes and alkylidenecyclopentanes.** J. Shantai, J. Herling and E. Gil-Av (Weizmann Inst. of Sci., Rehovoth, Israel). *J. Chromatography*, 1959, **2** (4), 406–410 (in English).—These compounds have been completely separated and determined on a stationary phase of ethanediol satd. with $AgNO_3$ at 30° with He as carrier gas. Retention volumes relative to toluene are given. G. BURGER

1439. **Identification of crystalline ferrocenes by X-ray diffraction.** W. L. Baun (Materials Lab., Wright Air Devel. Centre, Wright-Patterson Air Force Base, Ohio, U.S.A.). *Anal. Chem.*, 1959, **31** (8), 1308–1311.—Data on 22 crystalline ferrocenes are presented and a comparison is made between flat specimen diffractometer and Debye-Scherrer camera intensities. The intensities from the flat specimen did not originate from a random powder sample. K. A. PROCTOR

1440. **Absorption and fluorescence spectra of some mono- and di-hydroxynaphthalenes.** D. M. Hercules and L. B. Rogers (M.I.T., Cambridge, Mass., U.S.A.). *Spectrochim. Acta*, 1959, (6), 393–408.—A full discussion is given of the spectra of 1- and 2-naphthols and 1:3-, 1:4-, 1:5-, 1:6-, 2:3-, 2:6- and 2:7-dihydroxynaphthalenes, which have been recorded between 240 and 650 mμ in ethanolic soln. and in buffered and unbuffered soln. of known pH. E. G. CUMMINS

1441. **Ultra-violet absorption spectra of phthalic anhydride and related substances. Analytical method for naphthalene and 1:4-naphthaquinone.** H. Peters (Res. Lab., Reichhold Chem. A.-G., Hamburg, Germany). *Anal. Chem.*, 1959, **31** (8), 1326–1331.—Naphthalene (**I**) is determined by measuring the extinction at 311 mμ, 17 hr. after dissolving the sample in methanol. Owing to ester formation, phthalic anhydride makes almost no contribution to the extinction after this time. 1:4-Naphthaquinone (**II**) is determined by measuring the extinction at 347 mμ immediately after dissolving the sample. To avoid serious errors caused by photochemical changes, the substances must be stored in the dark and the work carried out in red light. The error for the determination of **II** is $\simeq$ $\pm$1%. The error for the determination of **I** is $\simeq$ $\pm$1% when the ratio of **I** to **II** in the sample is >10:1, while for 1 to 1 proportions the error is $\pm$3 to 5%. G. P. COOK

1442. **Preparative aspects of gas - liquid chromatographic separation. Quantitative determination of tetra-alkyltetrazenes.** E. M. Bens and W. R. McBride (Chem. Div., U.S. Naval Ordnance Test Station, China Lake, Calif.). *Anal. Chem.*, 1959, **31** (8), 1379–1383.—Three-, six- and ten-component mixtures are separated and determined by gas - liquid chromatography. Ultra-violet spectrophotometry is used to verify the separation and recovery of the various components. The relationship between peak-area and peak-height measurements with varied amounts of sample is discussed.

K. A. Proctor

1443. **Determination of manganese in gasoline by X-ray emission spectrography.** R. A. Jones (Res. Lab., Ethyl Corp., Detroit, Mich., U.S.A.). *Anal. Chem.*, 1959, **31** (8), 1341–1344.—Manganese, in the range 0·1 to 1·0 g per gallon of petrol, can be determined in about 15 min., with a standard deviation varying from 0·003 g per gallon at the 1 g per gallon level to 0·008 g per gallon at the 0·1 g per gallon level (6 determinations). Iron is used as an internal standard and the Fe to Mn intensity ratio of the sample is compared with ratios obtained with known standards. As the Fe to Mn intensity ratio is reasonably free from interferences, a single calibration curve can be used for all samples.

K. A. Proctor

1444. **Rapid determination of phosphorus in motor oils and additives.** J. E. Barney, II, J. G. Bergman and W. G. Tuskan [Standard Oil Co., Whiting (Indiana), U.S.A.]. *Anal. Chem.*, 1959, **31** (8), 1394–1396.—The sample is burned in O in a closed glass flask and the vapours are absorbed in dil. HNO_3. The P is then determined colorimetrically at 465 mμ as the molybdovanadophosphate complex. The precision is approx. the same as that of ASTM D 1091–54T. One sample can be analysed in 1 hr. G. P. Cook

1445. **Determination of the acidic constituents of coal utilisation products by titration in non-aqueous medium.** L. Kálmán and A. Újhidy (Res. Inst. for Heavy Chem. Ind., Veszprém, Hungary). *Nehézvegyipari Kutató Intézet Közleményei*, 1958, **1** (1–2), 129–136.—The acid content of coal-tar fractions is determined by potentiometric titration in dimethylformamide. Potassium methoxide dissolved in methanol and saturated with benzene is employed as titrant. The best results are obtained with a calomel - antimony or a calomel - glass electrode. The carboxyl group can be determined in the presence of phenols. The phenolic groups of mono- and poly-hydric phenols have the same dissociation constants, so that their sum can be titrated. Results from several fractions are tabulated. The error is $<0·4\%$. G. Szabo

1446. **Analysis of synthetic detergents.** W. B. Smith (Marchon Products Ltd., Whitehaven, Cumb., England). *Olii Min.*, 1959, **36** (5), 157–166.—A review, with 80 references.

L. A. O'Neill

1447. **Analysis of surfactant mixtures. I.** J. D. Knight and R. House (California Res. Corp., Richmond, U.S.A.). *J. Amer. Oil Chem. Soc.*, 1959, **36** (5), 195–200.—Surfactant mixtures are decomposed with 93% H_3PO_4 and the hydrophobic residues are identified by gas - liquid vapour-phase chromatography. The surfactant ($\simeq$5 g) is added to 250 ml of H_3PO_4 (b.p. 215°) and heated under reflux for 90 min. after the vapour temp. has reached 100°. After cooling, the mixture is transferred to a separating-funnel and sufficient acetone is added to give an aq. layer containing 70% of acetone, followed by the addition of 20% KOH soln. until alkaline. The mixture is then extracted with *iso*pentane (2 × 10 ml). The combined extracts are washed with water (2 × 10 ml) and filtered, and the neutral oil is obtained by removal of the solvent at 130° to 140° for 30 min. An acid oil fraction is obtained by acidifying the aq. acetone layer and extracting with *iso*pentane (2 × 10 ml), the extracts being washed with 3 *N* HCl (2 × 5 ml), filtered and the solvent removed. The methyl esters of this acid fraction are prepared by heating 100 mg of the sample under reflux with 25 ml of anhyd. methanol and 2 drops of conc. H_2SO_4 for 30 min., cooling and neutralising with 20% KOH soln. after diluting with 50 ml of water. The mixture is then extracted with *iso*pentane (2 × 10 ml) and the esters are recovered as described above. The neutral oils and esters are identified by gas chromatography. Three columns are used—(*i*) temp. 90°; 40 g of didecyl phthalate per 100 g of crushed brick (Johns-Manville C_{22}, 20 to 30 mesh) for the methyl*cyclo*pentane which is used as an internal standard; (*ii*) temp. 260°; 40 g of 85 to 100 penetration paraffinic asphalt per 100 g of crushed brick for the C_8 to C_{18} olefins, C_8 to C_{14} alcohols and commercial alkylates; (*iii*) temp. 300°; 40 g of silicone grease per 100 g of crushed brick for the methyl esters of fatty acids and the C_{14} to C_{18} alcohols. Good yields of the parent compounds are obtained with aromatic sulphonates, straight-chain alkyl sulphates, fatty acid amides and esters; dioctyl sulphosuccinate gives a mixture of octyl alcohols and olefins; the ethylene oxide adducts of dodecyl and tridecyl alcohols and *tert.*-dodecylmercaptan give olefins derived from the parent compound. The di*iso*butylene phenol - ethylene oxide adduct is decomposed into olefins, conjugated olefins, and alcohols derived from the breakdown of the aromatic ring. The alkane sulphonates do not give recoverable oils by this process.

G. R. Whalley

1448. **Paper-chromatographic separation of alkanolamines [in detergent powders].** E. Heinerth and J. Pollerberg. *Fette, Seif., Anstrichmitt.*, 1959, **61** (5), 376–377.—The method is suitable for the separation and identification of monoethanolamine (**I**), diethanolamine (**II**), triethanolamine (**III**) or other alkanolamines, *e.g.*, 1-aminopropan-2-ol (**IV**), and their salts. Alkanolamides must be hydrolysed before chromatography. *Procedure*—In order to distinguish between alkanol-amines and -amides, heat the ethanol extract ($\simeq$1 g) of the sample under reflux with 6 *N* HCl (30 ml) for 6 hr. Dilute the cooled soln. with H_2O (50 ml) and extract twice with ether. Evaporate the aq. phase to dryness, render it alkaline to phenolphthalein with 0·5 *N* alkali ($\not>$3 ml) and filter off the ppt. Spot the filtrate (2 to 5 μl) on S. & S. 2040b paper ($\simeq$7 cm × 30 cm) and chromatograph by the ascending method for 4 to 6 hr. with *iso*propyl alcohol - H_2O - conc. aq. NH_3 (40 : 9 : 1). When the solvent front has risen 15 to 20 cm, dry the paper overnight at room temp., or for 30 min. at 80°, and spray with 0·04% ethanolic bromocresol green (**V**): blue spots appear on a yellow background. For the identification, chromatograph a standard amine mixture ($\simeq$100 μg) on the same chromatogram. Primary amines (and hydrolysed primary alkanolamides) can also be detected, even after treatment with **V**, by spraying with a 0·2%

soln. of ninhydrin in *n*-butanol - 2 *N* acetic acid (19:1), $\not>$14 days old, followed by warming to 80° to 100°. Alkanolamines are detected similarly, but without the preliminary hydrolysis. Use $\simeq$100 μg of amine; the optimum quantity for each amine is 40 to 50 μg. The R_F values are—**I**, 0·59; **II**, 0·77; **III**, 0·86; and **IV**, 0·71. The sensitivity, when using **V**, is $\simeq$2 μg of amine; for monoalkanolamines, with ninhydrin, it is 0·2 μg. A. G. Peto

1449. **Use of ion exchangers for the separation of capillary-active substances, with special reference to cation-active and non-ionic compounds.** H. Hempel and H. Kirschnek (Farbenfabrik Bayer A.-G., Leverkusen, Germany). *Fette, Seif., Anstrichmitt.*, 1959, **61** (5), 369–374.—Activate the resin (0·2 to 0·4 mm mesh) by soaking it for 24 hr., then wash it alternately thrice with 3 to 4 times its own vol. of 10% HCl and thrice with 5% NaOH soln. Next, treat the cation exchanger with HCl and the anion exchanger with NaOH and finally wash the former with 5% methanolic HCl ($\simeq$200 ml) until the extinction of the eluate at 240 to 280 mμ is $<$0·02. Then wash the resin with H_2O or methanol till neutral. Fill the column (23 mm $\times$ 450 mm) to a height of $\simeq$140 mm with a suspension of the prepared resin in the solvent to be used subsequently. Just before use wash the column with H_2O (100 to 200 ml) or the solvent. To separate cationic from non-ionic substances on Lewatit CN 206 [H+] resin, pass a soln. of the substances (0·1 to 0·5 g) in H_2O (100 ml) through the column at a rate of 1 ml per min. and wash with H_2O ($\not<$100 ml) at 2 ml per min. Change the receiver and elute the non-ionic substance with methanol (150 to 200 ml) and the cationic component with the same vol. of 5% methanolic HCl. The elution curve is obtained by measuring the extinctions of 10-ml fractions at the max. of the first subsidiary band. The cationic constituent is determined by titration with sodium lauryl sulphate to bromophenol blue (Barr *et al.*, *J. Soc. Chem. Ind.*, 1948, **67**, 45). For the separation of anionic from non-ionic substances, again use Lewatit CN 206 [H+] resin, and the quantities already given. By washing with H_2O (600 ml), most of the anionic material is eluted, and the rest is obtained by elution with 5% acetic acid (150 ml). Wash with H_2O to neutrality and with methanol (200 ml) to remove the non-ionic component. The anionic component is determined by titration with Zephirol (benzalkonium chloride) by Barr's method. The non-ionic constituent is determined by u.v. spectrometry. A. G. Peto

1450. **Analysis of fluorescent brighteners.** L. E. Weeks, J. C. Harris and J. T. Lewis. *Soap, N.Y.*, 1959, **35** (5), 66–70, 277.—The function and mechanism of brighteners is discussed and the method of comparative testing employed is detailed, spectrophotometric analysis being used. A precision of $\pm$2% was attained. Chemical analysis is a further aid in characterisation of the components volatile at 105° and insoluble in ethanol, and in the determination of NaCl and organic S. The stability of brighteners to NaOCl is likewise informative. S.C.I. Abstr.

1451. **Application of gas chromatography to the identification of some terpenes present in the essential oils from conifers.** H. Cvrkal and J. Janák (Forestry Research Institute, Opočno). *Coll. Czech. Chem. Commun.*, 1959, **24** (6), 1967–1974 (in German).—Elution volumes are given for some terpenes on dimethylpolysiloxane at 140° and 180° with H as carrier gas. Results are quoted which show the variations in the monoterpene hydrocarbons present in pine oil (*Pinus silvestris* L.) from different sources, and by a combination of fractional distillation and gas chromatography it was shown that pine oil from *Picea excelsa* L. contained at least 23 components. W. T. Carter

1452. **Investigation and analysis of some cosmetics [hair dyes].** C. Iacobelli Turi (Ist. Sup. Sanità, Roma). *Ann. Chim., Roma*, 1959, **49** (3), 459–498.—The identification of hair dyes in commercial preparations is described, and a small and convenient chromatographic apparatus is illustrated. The procedure involves a preliminary removal of oily matters from creams, shampoos, etc., by methods that do not alter the dyes, which are then extracted with diethyl ether from the soln. (*i*) slightly acidified with HCl, then (*ii*) rendered alkaline with NaOH soln. containing $NaHSO_3$ and finally (*iii*) faintly acidified with HCl and treated with $NaHCO_3$. These extracts contain, respectively, phenols, amines and aminophenols. Each fraction is examined by one-dimensional chromatography on S. & S. paper No. 2043 Mgl, with *n*-butanol - acetic acid - water (4:1:5), or by two-dimensional chromatography with *n*-butanol - ethanol - water (4:1:5) as the second solvent. The dyes are identified by their R_F values, their reactions on the paper with various reagents, and by comparison with standard spots. R_F values for the two solvent systems are listed for 26 dyes, and their reactions with 11 reagents are illustrated by colour plates. The detailed application of the procedure to 23 commercial samples is described. C. A. Finch

1453. **Identification of organic compounds. XXIX. The paper chromatography of di- and triphenylmethane dyes.** J. Gasparič and M. Matrka (Research Inst. Organic Synthesis, Pardubice-Rybitví). *Coll. Czech. Chem. Commun.*, 1959, **24** (6), 1943–1949 (in German).—The dyes are separated by descending chromatography on Whatman No. 3 paper impregnated with *n*-dodecanol. R_F values are given for ethanol - aq. NH_3 - water (2:2:1), ethanol - aq. NH_3 (1:1) and ethanol - 5% KCl soln. (1:1) as mobile phases. The separated dyes are detected by treating the dried paper with acetic acid or HCl vapour, and leuco bases are detected by spraying with 1% $K_2Cr_2O_7$ soln. - 1% oxalic acid soln. (1:1). The relationship between R_F values and structure is discussed. W. T. Carter

1454. **Isolation and estimation of 2-naphthol in D & C Red 36 [C.I. Pigment Red 4].** L. Koch (H. Kohnstamm and Co., Inc., Brooklyn, N.Y., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1959, **42** (2), 444–445.—The pigment (5 g) is stirred mechanically with ethanol (90 ml) and 10% aq. NaOH soln. (10 ml) for 30 min. The mixture is then poured into water, treated with *p*-nitrophenyldiazonium chloride, and examined spectrophotometrically against a standard at 490 mμ. The extraction procedure employed is much more rapid than extraction with *iso*propyl alcohol in a Soxhlet apparatus, and gives equally accurate results. A. A. Eldridge

1455. **Colorimetric determination of methylcellulose with diphenylamine.** G. Kanzaki and E. Y. Berger (Goldwater Memorial Hosp., Welfare Island, New York, U.S.A.). *Anal. Chem.*, 1959, **31** (8), 1383–1385.—Methylcellulose (**I**) reacts with an acid soln. of diphenylamine on heating to give a colour which follows Beer's law at 640 mμ in the range from 10 to 100 μg of **I** per ml. The reaction is sensitive to temp., which must be constant

to within 0·1° during heating for a given set of conditions. The amount of reagent, the temp. and time of heating may be varied within limits to obtain a desired extinction coefficient. The precision is ±2% from 158 determinations on a standard sample. G. P. COOK

1456. **Infra-red determination of nitrocellulose n mixtures of cellulose resins.** H. M. Rosenberger and C. J. Shoemaker (A. B. Dick Co., Chicago, Ill., U.S.A.). *Anal. Chem.*, 1959, **31** (8), 1315–1317.—Nitrocellulose is determined in the presence of other coatings components, *e.g.*, cellulose esters and ethers and alkyd resins, by the characteristic 11·92-μ absorption band. A maximum error of ±1% and a mean error of ±0·4% were obtained from 4 determinations on each of 14 synthetic mixtures having nitrocellulose contents in the range from 1 to 80%. G. P. COOK

1457. **Determination of silver in photographic papers and films by wet ashing.** E. E. Gansel (Ansco Res. Dept., Binghampton, N.Y., U.S.A.). *Anal. Chem.*, 1959, **31** (8), 1366–1368.—Silver ($\simeq$12 mg) in photographic papers and films is determined by potentiometric titration with 0·001 to 0·05 N KBr of the Ag_2SO_4 produced by wet ashing of the sample with HNO_3 and H_2SO_4. The accuracy is within ±2%. J. P. STERN

1458. **Spot-testing urethane polymers.** Mobay Chemical Co. *Canad. Paint Varn.*, 1959, **33** (5), 50–51.—A rapid test for differentiating between polyester- and polyether-based urethane polymers is described. *Procedure*—A few drops of 2 N methanolic KOH containing phenolphthalein are placed on the surface of the polymer, and partly neutralised with a satd. soln. of hydroxyammonium chloride. (Highly cross-linked polymers may require heating for 20 to 40 sec. at $\not>$50° at this stage.) After $\not<$10 sec. the spot is acidified with N HCl and a few drops of $FeCl_3$ soln. are added. A violet colour indicates a polyester-based urethane polymer. With polyester products derived from dimerised fatty acids, the colour is brownish rather than violet. With polyether-based polymers, only the yellow colour of $FeCl_3$ is observed. D. R. DUNCAN

1459. **Paper-chromatographic analysis of dicarboxylic acids in polyesters.** P. Fijolka, R. Kayler and I. Lenz (Inst. für Kunststoffe der Dtsch. Akad. der Wissenschaften, Berlin). *Kunststoffe*, 1959, **49** (5), 222–225.—Saponification of linear polyesters is best effected with aqueous, not ethanolic, alkali to prevent formation of ethoxysuccinic and malic acids from fumaric and maleic acids. The ester (0·3 g) in 25 ml of acetone is saponified with 0·5 N aq. NaOH in 20% excess, and neutralised with 0·5 N aq. HCl, and the mixture is passed through a cation-exchange column. The acids are recovered by washing the column with 150 ml of ethanol, the vol. is reduced to 50 ml by distillation, and 0·01 to 0·02 ml is chromatographed on paper, with the organic phase of benzene - acetone - formic acid - water (5:5:1:3) as developer. To identify the acids in the presence of the ester, the mixture is chromatographed with the two-phase solvent, the paper being immersed in the aqueous phase. Data are recorded for model compounds and mixtures. J. L. PROSSER

1460. **Separation of poly(vinyl chloride) and poly(vinyl acetate) by chromatographic methods.** W. J. Langford and D. J. Vaughan (Tufnol Ltd., Perry Barr, Birmingham, England). *Nature*, 1959, **184**, 116–117.—Separation of the mixed polymers in *iso*butyl methyl ketone soln. was achieved on a column of activated charcoal saturated with *iso*butyl methyl ketone, the poly(vinyl chloride) being the least strongly adsorbed. It was also possible to separate these substances on Whatman No. 4 paper with the same solvent. The acetate spot remained stationary. B.D.H. Universal Indicator diluted with an equal vol. of water was sprayed on to the paper to locate the spots. G. S. ROBERTS

1461. **Methods of testing plastics. Part 4: Analytical methods and viscosity in solution.** British Standards Institution (2 Park St., London). Amendment No. 3 to B.S. 2782: Part 4: 1958 (published 12.6.59).—A correction to the analytical procedure is made.

1462. **Chromatography of the sulphonation products of phenol - formaldehyde condensates. II. Quantitative determination of phenolsulphonic acids.** F. Langmaier, E. Mück and D. Kokeš (Research Inst. for the Leather Industry, Otrokovice). *Coll. Czech. Chem. Commun.*, 1959, **24** (6), 2066–2067 (in German).—The method described in Part I (*cf.* Mück *et al.*, *Anal. Abstr.*, 1959, **6**, 616) can be made quant. by eluting the *o*- and *p*-phenolsulphonic acids from a duplicate chromatogram with ethanol, and measuring the extinctions of the resulting soln. at 280 and 274 mμ, respectively; concn. of phenolsulphonic acids in the range 0·005 to 0·06 mg per ml can be determined with an accuracy of $\simeq$5%. W. T. CARTER

1463. **Determination of *p-tert.*-butylbenzoic acid in coconut oil-type modified alkyds.** P. J. Secrest and B. Kosciesza (Sherwin-Williams Co., Chicago, Ill., U.S.A.). *Anal. Chem.*, 1959, **31** (8), 1402–1404.—The method is based on the differential measurement of extinction at 282·5 and 300 mμ. The procedure is applicable only to alkyd resins containing coconut-type and similar acids, which are only slightly unsaturated and exhibit only a slight difference in extinction at the analytical wavelengths. The accuracy is dependent on the degree of unsaturation of the acids present. G. P. COOK

1464. **Identification and analysis of polyurethane rubbers by infra-red spectroscopy.** P. J. Corish (Chem. Res. Dept., Fort Dunlop, Birmingham, England). *Anal. Chem.*, 1959, **31** (8), 1298–1306.—A qual. method is described for a wide range of polyurethanes. For those containing polyesters, the acid in the ester wax and the di*iso*cyanate can in most cases be identified by the direct i.r. examination of stretched microtomed sections of the rubber. A method of hydrolysis and separation is used for the identification of the glycol used in the ester wax. In some cases, examination of the hydrolysis products was needed to identify the original di*iso*cyanate. G. P. COOK

1465. **Analysis of water emulsion paints.** M. H. Swann and G. E. Esposito (U.S. Army Ordnance Coating & Chem. Lab., Aberdeen Proving Ground, Md.). *Off. Dig. Fed. Paint Varn. Prod. Cl.*, 1959, **31**, 624–628.—Non-volatile matter (pigment and binder) is determined gravimetrically by drying the paint sample to constant weight in an oven at a temp. below that at which the polymer present decomposes. The residue may be pyrolysed and the i.r. absorption spectrum of the distillate is used for the identification of the polymer (Harms, *Anal. Abstr.*, 1954, **1**, 125). The procedure for

determining the pigment content of paints based on an acrylic copolymer or poly(vinyl acetate) latex is as follows. A sample (0·5 to 1·0 g) is weighed into a pan of aluminium foil (diam. ≮ 60 mm) and allowed to dry in the air at room temp. for 24 hr. The film is stripped off and heated under reflux for 1 hr. with 25 ml of benzene and well stirred, either during the extraction or subsequently. The sample is then washed into a centrifuge tube with benzene. After centrifuging and pouring off the supernatant liquid, the process is repeated twice, with ethyl acetate as suspending agent. The tube with pigment is then dried at 105° and weighed. With butadiene - styrene copolymer latex paints, the following modified procedure must be used. Approximately 10 ml of paint is thinned with 5 ml of water and spread on a glass plate with a Bird film applicator of 0·001 or 0·0015 in. depth. It is dried at 150° for 2 hr. or (if this causes decomposition) at 60° for 4 hr. A weighed portion of the stripped-off film is heated under reflux for 4 hr. with *o*-dichlorobenzene and then centrifuged (with acetone for the transferring and washing) and dried and weighed as before. The pigment sample so obtained may be analysed by semi-micro methods.

D. R. Duncan

1466. **Hydroxyproline as a measure of hide substance.** J. H. Bowes (British Leath. Mnfrs. Res. Assoc., Egham, Surrey). *J. Soc. Leath. Tr. Chem.*, 1959, **43** (6), 203–211.—A method of estimating collagen by determining the hydroxyproline (**I**) obtained by acid hydrolysis is based on the Neuman and Logan technique (*cf. Brit. Abstr. C*, 1950, 412) involving the oxidation of **I** with H_2O_2, followed by colour development with *p*-dimethylaminobenzaldehyde. *Procedure*—To 25-ml flasks are added, in order, the unknown soln. (2 ml), 0·05 *M* $CuSO_4$ (2 ml) 1·25 *N* NaOH (2 ml) and 6% H_2O_2 (2 ml). Four standards (10, 20, 30 and 40 µg of **I**) and a blank are also set up. The H_2O_2 must be added rapidly and the flask shaken once every min. for 5 min. The H_2O_2 is then destroyed by heating to 40° for 15 min. The flasks are cooled rapidly in water and 1·8 *N* H_2SO_4 (10 ml) and 5% *p*-dimethylaminobenzaldehyde in propanol (5 ml) is added. The pink colour is developed at 70° for 30 min., the flasks are cooled, made up to volume with H_2O and the extinction is read after 15 min., against a blank, with a Spekker absorptiometer and 2-cm cells. The derivation of a factor for calculation, and the interference of Ca and vegetable tanning materials, are discussed.

C. A. Slater

See also Abstracts—**1234**, Xanthate S in viscose. **1273**, Silver in photographic emulsions. **1325**, Zirconium in paint driers. **1553**, Determination of 4-aminosalicylic acid, and 3- and 4-aminophenol. **1578**, Polyoxyethylene glycols in edible fats. **1584**, Acetic esters in air. **1591**, Separation of pentoses and hexoses. **1610**, Gas chromatography of hydrocarbons.

4.—BIOCHEMISTRY
INCLUDING DRUGS, FOOD, SANITATION, AGRICULTURE

Biological fluids, animal and vegetable tissues

1467. **Flame-photometric determination of sodium, potassium and calcium in serum and urine with special consideration of errors due to interference.** A. Häussler and P. Hajdú (Pharm.-Wissenschaftl. Lab. d. Farbwerke Hoechst A.-G.). *Arch. Pharm., Berlin*, 1959, **292** (5); *Mitt. dtsch. pharm. Ges.*, 1959, **29** (5), 73–84.—Procedures are described. The presence of Na affects the intensity of the K and Ca lines and corrections for Na are, therefore, introduced in the calculation of the results. Other corrections are made for protein. For Ca a method is adopted in which known amounts of Ca are added to the test soln. to eliminate the considerable error due to the presence of protein.

M. H. Sawistowska

1468. **Use of the cobaltinitrite method for the determination of potassium in plant sap.** B. A. Lebedev. *Byull. Nauch.-Tekh. Inform. Ural'sk. Nauch.-Issled. Inst. Sel'sk. Khoz.*, 1958, (4), 39–40; *Ref. Zhur., Khim.*, 1959, (18), Abstr. No. 64,096.—A new modification of the cobaltinitrite method has been developed for use under field conditions. *Procedure*—Place 0·05 g of $Na_3Co(NO_2)_6$ in each of 5 identical tubes (1 × 6 or 8 cm); to the first four add known amounts of a potassium soln. and to the fifth add 2 drops of the sap. Then to each tube add 3 ml of boiled water, mix to attain complete dissolution, and compare the degree of cloudiness in the tubes. The method is less sensitive than the hexanitrodiphenylamine method. C. D. Kopkin

1469. **Determination of gold in biological materials by neutron activation analysis.** D. Gibbons (Isotope Div., A.E.R.E., Harwell, England). *Int. J. Appl. Radiation Isotopes*, 1958, **4** (1–2), 45–49.—The method has been applied to human and animal liver and pleural fluids. Samples (1 g) were irradiated together with standards of $AuCl_3$ soln. Two procedures for the purification of the ^{198}Au are described. In the first, which has a reproducibility of ±2%, the gold carrier with the ^{198}Au is repeatedly pptd. as the metal and is extracted into ethyl acetate; β-counting is used to measure the activity. The second procedure, which is more rapid, involves a much simplified chemical separation and the measurement of the 0·41-MeV γ-radiation by γ-spectrometry; results are accurate to within ±10%.

G. J. Hunter

1470. **Determination of magnesium in blood serum by atomic absorption spectroscopy.** J. B. Willis (Div. of Chem. Phys., C.S.I.R.O. Chem. Res. Lab., Melbourne, Australia). *Nature*, 1959, **184** (Suppl. No. 4), 186–187.—Serum is diluted 1 to 20 with a 1% soln. of EDTA (disodium salt), and is sprayed into an air - gas (acetylene or coal gas) flame of the Lundegårdh type. The absorption of the Mg resonance line at 2852 Å is measured, calibration being made with soln. containing 0·05 to 0·15 mg of Mg per 100 ml. Recovery of added Mg was 100 to 102% and duplicate readings seldom differed by more than 2%.

H. F. W. Kirkpatrick

1471. **Direct micro-complexometric analysis of calcium in biological materials.** K. Mori (Montreal Univ., Canada). *Arch. Biochem. Biophys.*, 1959, **83** (2), 552–562.—The method is based on the direct titration of the Ca^{2+} with the disodium salt of 1 : 2-diamino*cyclo*hexane-NNN′N′-tetra-acetic acid (**I**) at pH 12, with calcein as indicator. *Procedure*—Add 0·1 ml of serum or plasma to 5 ml of 3 *N* KOH in a small beaker. Titrate with **I** to a pink non-fluorescent end-point. Deduct the blank. Urine requires preliminary dilution, and tissues

homogenisation with 15 ml of 10% trichloroacetic acid soln. followed by centrifugation. Carry out the assay on 1 to 2 ml of the clear supernatant liquid. W. H. C. SHAW

1472. **Rapid and accurate automatic titration method for determination of calcium and magnesium in plant material with EDTA titrant.** H. V. Malmstadt and T. P. Hadjiioannou (Dept. of Chemistry and Chemical Engng, Univ. of Illinois, Urbana, U.S.A.). *J. Agric. Food Chem.*, 1959, **7** (6), 418–420.—In the method described, phosphate is removed from plant material, after wet digestion, by pptn. with zirconium nitrate. Calcium and Mg are extracted with CCl_4 as the diethyldithiocarbamate complexes. The Sargent Spectro-Electro automatic titrator is used, with standard EDTA (disodium salt) soln., to determine Ca plus Mg (indicator Eriochrome black T), and Ca [indicator Calcon (C.I. Mordant Black 17) at pH 13. Triethanolamine may be added before the titration to mask traces of other metals remaining in solution, but in most cases this is not necessary. M. D. ANDERSON

1473. **Estimation of arsenic in biological tissue by activation analysis.** H. Smith (Dept. of Forensic Med., Univ. of Glasgow, Scotland). *Anal. Chem.*, 1959, **31** (8), 1361–1363.—The sample is irradiated in an atomic pile and then digested with HNO_3 - H_2SO_4 (5:3). A Gutzeit separation is then combined with a determination with a Geiger tube which accepts liquid samples. Blanks are not required. A single hair weighing 0·5 mg can be analysed, and 2 operators can analyse up to 100 samples in 2 days. The results agree well with those obtained by other methods. K. A. PROCTOR

1474. **Determination of arsenic, gold and cobalt in biological tissues.** B. McS. Dale (Ohio State Univ., U.S.A.). *Dissert. Abstr.*, 1959, **20** (2), 472–473.—The determinations on tissue samples from rats and mice bearing transplanted tumours were carried out by ashing the samples, wrapping the ash in aluminium foil, and irradiating with neutrons in a nuclear reactor for 1 week, together with standards of Au, As and Co. Half-life measurements were used to identify the activities of Au and As, and to check for radioactive contamination, and for Co this was done by absorbing the β-rays in aluminium, and measuring the γ-spectrum in a scintillation spectrometer. Quantities were calculated by comparing counting rates with those of the standards. Gold was separated by extraction of an acid solution with ethyl acetate, and plating on copper from a basic cyanide solution; the error in determining known amounts of Au was $\not>$9·7%. Arsenic was separated by pptn. as the sulphide, and purified by distillation as $AsCl_3$; the error was $\not>$12·3%. Cobalt was separated by pptn. in a basic solution as the sulphide, and re-pptg. as the 1-nitroso-2-naphthoxide; the error was $\not>$5·1%. M. D. ANDERSON

1475. **Spectrophotometric determination of vanadium in plant materials.** G. B. Jones and J. H. Watkinson (Div. of Biochem., C.S.I.R.O., Univ. of Adelaide, S. Australia). *Anal. Chem.*, 1959, **31** (8), 1344–1347.—After electrolytic removal of Fe and other interfering ions and extraction of V as tetramethylenedithiocarbamate, V (<10 μg) is determined absorptiometrically at 450 mμ as the benzohydroxamate in octanol - CCl_4 soln. The mean coeff. of variation is 3% for <1 μg of V and 0·5% for 3 to 10 μg of V. The method is applicable to soil samples. *Procedure*—Digest the sample (5 to 15 g) with HNO_3 and then excess of $HClO_4$, filter off any siliceous residue, and electrolyse the filtrate at 6 V and 4 to 5 amp. for 1 hr. Add bromine water and boil off the excess of Br. Add 30% H_2O_2 (4 drops) to the cooled soln. and extract with 2% aq. ammonium tetramethylenedithiocarbamate soln. and $CHCl_3$ until the $CHCl_3$ extract is colourless. Evaporate the extract to dryness and decompose the organic residue with conc. HNO_3 (1 ml) and conc. H_2SO_4 (9 drops). Add H_2O (10 ml) and bromine water and boil off the excess of Br. Add 10% ammonium acetate soln. (1 ml) and adjust the pH to 4·6 (bromophenol blue) with 20% aq. NH_3. Dilute to 50 ml, add 0·1 *M* benzohydroxamic acid (2 ml) and, after 5 min., extract by shaking for 2 min. with a measured vol. of octan-2-ol - CCl_4 (1:3). Measure the extinction of the extract at 450 mμ against a reagent blank. J. P. STERN

1476. **Spectrophotometric determination of the oxygen saturation of blood.** G. W. Johnston, F. Holtkamp and J. R. Eve (Fitzsimons Army Hosp., Denver, Colo., U.S.A.). *Clin. Chem.*, 1959, **5** (5), 421–425.—A simpler sampling technique is described for use with the method of Gordy and Drabkin (*J. Biol. Chem.*, 1957, **227**, 285). H. F. W. KIRKPATRICK

1477. **Micro-determination of sulphur in plant material.** O. Iismaa (C.S.I.R.O. Reg. Pastoral Lab., Armidale, N.S.W.). *J. Aust. Inst. Agric. Sci.*, 1959, **25** (2), 136–138.—The combustion method proposed by Schöniger (*Mikrochim. Acta*, 1955, 123; 1956, 869) has been modified. The sample (20 to 150 mg), wrapped in filter-paper, is ignited in a platinum spiral in O contained in a 1-litre Erlenmeyer flask and in the presence of 10 ml of absorbing soln. (0·4 *N* KOH and 3 drops of 30% H_2O_2). After shaking and setting aside for 40 min., an aliquot containing 20 to 70 μg of S is boiled for a few minutes, acidified with HCl and evaporated to dryness to remove NO_3^-. Sulphate is determined by the methylene blue method of Johnson and Nishita (*cf. Brit. Abstr. C*, 1952, 356). E. G. BRICKELL

1478. **The determination of iron and copper in biological fluids after therapy with EDTA.** V. Fišerová-Bergerová (Inst. Hyg. and Ind. Diseases, Prague). *Pracovní Lékařství*, 1958, **10** (1), 25–27.—Iron and Cu can be determined colorimetrically in the presence of EDTA, Fe with 2:2′-dipyridyl and Cu with Na diethyldithiocarbamate. The method of Ventura and White (*Analyst*, 1954, **79**, 39) was used for blood and was modified for use with urine without previous ashing. *Procedure for Fe and Cu in urine*—To the sample (30 ml) add a few drops of aq. NH_3 and 2 drops of H_2O_2 (30%) and evaporate to one-half of the volume. Cool, repeat the addition of aq. NH_3 and H_2O_2 and evaporate to 3 ml. Cool, add 6 *N* HCl (2 ml), transfer to a centrifuge tube, add trichloroacetic acid (20%) (3 ml), dilute to 8 ml with twice-distilled H_2O and centrifuge. To the supernatant liquid (6 ml) add hydroxyammonium chloride soln. (2%) (3 ml), and neutralise exactly with aq. NH_3 or HCl to phenolphthalein; dilute with ammonium acetate soln. (25%) to 17 ml, mix, and to 8 ml of the soln. add 2:2′-dipyridyl soln. (0·5% ethanolic) (1 ml). After 24 hr. measure the extinction at 440 mμ. Extract another portion of the sample, neutralised to phenolphthalein, with a mixture of diethyl ether (10 ml) and an ethanolic

soln. of Na diethyldithiocarbamate (2%) (5 ml), separate the ether layer, wash with twice-distilled H_2O, dilute with ethanol to 10 ml and measure the extinction. The average error is $\simeq \pm 10\%$.

J. Zýka

1479. **Spectroscopic identification of α-emitting nuclides in biological material.** W. V. Mayneord and C. R. Hill (Physics Dept., Inst. Cancer Res., Royal Cancer Hosp., London). *Nature*, 1959, **184**, 667–669.—The α-activity of normal biological materials was determined by measuring the size of individual impulses caused by the electrons produced by the action of the energy of the α-particles on the ionisation of an electron-free gas. The constructional details of the ionisation chamber are given. This is used in conjunction with EKCO 1049B head and main amplifiers and a CDC 100-channel pulse-height analyser. Analysis of a 100% whole wheat cereal has given an α-activity of 32×10^{-13} C per g of ash. It also showed the presence of ^{226}Ra and ^{228}Th and their daughter elements. The α-activity of British grass samples ranged from 1·0 to 170×10^{-13} C per g of ash. Most of the activity was of small energy range; ^{210}Po accounts for about 90% of the total α-activity. The method is not sufficiently sensitive for the analysis of normal bone.

S. Baar

1480. **Determination of carbonate in biological solids.** R. F. Sharp, J. S. Elliot and L. Lewis (Fairmont Hosp., San Leandro, Calif., U.S.A.). *Clin. Chem.*, 1959, **5** (5), 488–491.—The material is ground in a mortar, and carbonate is extracted with 0·1 *M* EDTA. The determination is then carried out in a Van Slyke - Neill manometric apparatus.

H. F. W. Kirkpatrick

1481. **Clinical applications of infra-red spectroscopy. Analysis of renal tract calculi.** M. Weissman, B. Klein and J. Berkowitz (Vet. Admin. Hosp., Bronx, N.Y., U.S.A.). *Anal. Chem.*, 1959, **31** (8), 1334–1338.—The absorption spectra of types of calculi usually found are compared with those of the pure substances and of mixtures. Qualitative identification and semi-quantitative determination of components are facilitated.

H. F. W. Kirkpatrick

1482. **Post-exposure analysis of organic compounds in the blood by a rapid infra-red technique.** R. D. Stewart, D. S. Erley, T. R. Torkelson and C. L. Hake (Med. Dept., The Dow Chemical Co., Midland, Mich., U.S.A.). *Nature*, 1959, **184** (Suppl. No. 4), 192–193.—Oxalated blood (10 ml) is extracted with CS_2, dichloromethane or other suitable solvent (15 ml) by end-over-end inversion for 5 min., and the tube is then centrifuged at 500 *g* for 10 min. in a refrigerated centrifuge. The solvent layer is transferred to a standard infra-red cell and scanned from 2 to 16 μ. The method is applicable to urine and probably to other biological fluids. Results after exposure to benzene and trichloroethylene, and after ethanol and *iso*propyl alcohol taken orally, are tabulated.

H. F. W. Kirkpatrick

1483. **Spectrophotofluorimetric assay of griseofulvin.** C. Bedford, K. J. Child and E. G. Tomich (Glaxo Laboratories Ltd., Greenford, Middlesex, England). *Nature*, 1959, **184** (Suppl. No. 6), 364–365.—To determine 0·05 to 0·5 μg per ml of griseofulvin (I) in blood, plasma, serum or urine, shake the sample (<1 ml) with 1% ethanol (1 ml) and diethyl ether (10 ml) for 15 sec. Evaporate to dryness an 8-ml aliquot of the ether phase, dissolve the residue in 1% ethanol (10 ml) and measure in a spectrophotofluorimeter at 450 mμ with an activating wavelength of 295 mμ against a standard soln. of **I** (0·5 μg per ml) at the same temp. The fluorescence is independent of pH in the range 3 to 10, but decreases markedly with rise of temp. Include pre-dosage samples of the biological fluid, with and without added **I**, in each set of assays. Blank urine values vary greatly, and to minimise this effect the pH of a urine sample should be raised to about 10 before extraction with ether. The mean percentage recovery of **I** added to heparinised blood at concn. in the range 1 to 5 μg per ml was 96, with a standard error of ±1·4 (25 assays); the percentage recovery from urine was 90 ±2·9 (30 assays).

A. R. Rogers

1484. **Spectrophotometric determination of 3-o-tolyloxy-1 : 2-propanediol [3-(2-methylphenoxy)-propane-1 : 2-diol] (mephenesin) and its metabolite in plasma and urine.** A. R. Maass, P. L. Carey and A. E. Heming (Smith Kline and French Lab., Philadelphia, Pa., U.S.A.). *Anal. Chem.*, 1959, **31** (8), 1331–1334.—*Procedure for plasma*—The sample (10 ml) is extracted with ether (4 × 10 to 20 ml), by gentle shaking, any emulsion being broken by centrifuging or adding NaCl. The ether extract is concentrated to 10 ml and assayed for mephenesin (**I**) by its extinction at 300 and 270 mμ. The plasma is adjusted to pH 3 with dil. HCl and the ether extraction is continued to remove 3-(2-methylphenoxy)lactic acid (**II**). The pooled ether extracts, collected over 10 ml of 0·1 *N* NaOH, are concentrated to 10 ml, and shaken with the alkali, and the extinction of each layer is read at 300 and 270 mμ. **II** is identified by its absorption pattern in the dil. alkali and its concn. is calculated from the reading at 270 mμ. *Procedure for urine*—The sample (5 ml) is diluted with H_2O (20 ml), the pH is adjusted to 7·5 to 8·5 and **I** is extracted with ether as for plasma, the pooled ether extracts being collected over 0·1 *N* NaOH (5 ml). The ether is concentrated to 10 ml and shaken with the alkali, and the extinction of each layer is measured at 270 mμ. (Some **II** may be present in the alkali.) **II** is then determined by adjusting the dil. urine to pH 3, exhaustively extracting with ether, pooling the extracts over 40 ml of 0·1 *N* NaOH, and completing the determination as for plasma.

H. F. W. Kirkpatrick

1485. **Micro-colorimetric method for the determination of sorbitol, mannitol and glycerol in biological fluids.** J. M. Bailey (Johns Hopkins Med. Sch., Baltimore, Md., U.S.A.). *J. Lab. Clin. Med.*, 1959, **54** (1), 158–162.—In the method described, the deproteinised sample (0·1 ml) containing 5 to 50 μg of sorbitol, mannitol or glycerol is treated with an acid solution of HIO_4 for a limited time to minimise the oxidation of glucose. The excess of HIO_4 is then destroyed with aq. $NaAsO_2$ soln. and the formaldehyde is determined colorimetrically with phenylhydrazine and $K_3Fe(CN)_6$. Blood-serum components do not interfere. The method may be modified for the determination, on one sample, of glucose (by a copper reduction procedure) and the polyhydric alcohols.

W. H. C. Shaw

1486. **Improved diacetyl monoxime method for the determination of urea in blood and urine.** Motoshi Kitamura and Iwas Iachi (Tokyo Hosp. of Japan Monopoly Corp., Japan). *Clin. Chim. Acta*, 1959, **4** (5), 701–706 (in English).—In the proposed

method, $HClO_4$ is used as oxidising agent and H_2SO_4 as solvent. *Procedure*—Add 0·1 ml of serum and 1·4 ml of 10% trichloroacetic acid soln. to 1·5 ml of H_2O, mix and centrifuge. To 1 ml of filtrate in a stoppered tube add 1 ml of 1% diacetyl monoxime soln. in 5% acetic acid and 4 ml of $HClO_4$ - H_2SO_4 reagent (60 ml of conc. H_2SO_4 is added to 240 ml of H_2O with cooling, followed by 22 ml of 70% $HClO_4$). Heat in boiling water for 30 min., then cool in running water and read the colour at 470 mμ. A calibration curve is used for evaluation of the urea. H. F. W. KIRKPATRICK

1487. **Spectrophotometric determination of uric acid in serum and urine with the aid of uricase.** S. K. Wadman (Klin.-chem. Lab., Zuiderziekenhuis, Rotterdam, Netherlands). *Pharm. Weekbl.*, 1959, **94** (13), 425–431.—The method of Praetorius (*Scand. J. Clin. Lab. Invest.*, 1949, **1**, 222; 1953, **5**, 273) is slightly modified. *Procedure for serum*—In a 1-cm cell mix 3·00 ml of 0·0667 *M* glycine buffer (5·0 g of glycine, 3·9 g of NaCl and 0·55 g of NaOH per litre) with 0·02 ml of uricase soln. (1 ampoule of uricase "Leo" dissolved in 0·5 ml of glycine buffer) and measure the extinction (A_1) at 293 mμ against a blank of glycine buffer. Mix 2·90 ml of glycine buffer with 0·10 ml of serum and measure the extinction (A_2) at 293 mμ against a mixture of 3·00 ml of glycine and 0·08 ml of serum. Add 0·02 ml of uricase soln. to the soln. which contains 0·10 ml of serum, mix and measure the extinction (A_3) when it has become constant. Then $\triangle A (= A_1 + A_2 - A_3)$ is a measure of the uric acid content. A calibration graph is prepared with a standard uric acid soln. (10 mg per 100 ml of glycine buffer). For 20 μg of uric acid the reaction time is $\not>$20 min.; for 50 μg of uric acid this time increases to 45 min. For economy of time and enzyme the sample should contain $\not>$25 μg of uric acid. *Procedure for urine*—Dilute the urine 10 times and follow the procedure as for serum. In the determination of A_2 it is not necessary to add 0·08 ml of urine to the blank soln. M. J. MAURICE

1488. **Occurrence and quantitative determination of 2-dimethylaminoethanol in animal-tissue extracts.** C. G. Honegger and R. Honegger (Dept. of Pharmacol., Div. of Basic Health Sciences, Emory Univ., Atlanta, Ga., U.S.A.). *Nature*, 1959, **184** (Suppl. No. 8), 550–552.—Extract unbound amines from the homogenised tissues with 80% ethanol at pH 2 or 3. Concentrate the extract under reduced pressure, saturate it with $Ba(OH)_2$ and steam-distil under nitrogen at a pressure of 15 to 25 torr; collect the distillate in *N* HCl in fractions and remove excess of HCl under reduced pressure. To liberate and separate bound amines, heat under reflux the residue from the steam-distillation and the ethanol-insoluble ppt. for 6 hr. with a saturated aq. soln. of $Ba(OH)_2$, extract the hydrolysate with 80% ethanol at pH 2 and fractionate as described above. Determine 2-dimethylaminoethanol (**I**) in the fractions by chromatography on a 5-ft. column of Carbowax 20M on Chromosorb, pretreated with methanolic NaOH soln., with He as carrier gas at 125°. As little as 0·1 to 0·5 μg of **I** can be detected and quantities of $>$20 μg can be determined with an error of $<$2%. Paper electrophoresis and paper chromatography have been used to confirm the identity of the amines. A. R. ROGERS

1489. **Concentration of hyaluronic acid in synovial fluid.** B. Decker, W. F. McGuckin, B. F. McKenzie and C. H. Slocumb (Mayo Clinic and Mayo Found., Rochester, Minn., U.S.A.). *Clin. Chem.*, 1959, **5** (5), 465–469.—A modification of the Dische carbazole method for the determination of uronic acids (*J. Biol. Chem.*, 1947, **167**, 189) is used. The results confirm values reported by other methods, and suggest an inverse relationship between the hyaluronic acid concn. and the degree of synovitis. H. F. W. KIRKPATRICK

1490. **Improved test for phenylketonuria.** C. O. Rupe and A. H. Free (Miles-Ames Res. Lab., Elkhart, Ind., U.S.A.). *Clin. Chem.*, 1959, **5** (5), 405–413.—A strip test (Phenistix) is described. A cellulose strip is impregnated with Fe^{3+} as a colour-producing reagent, Mg^{2+} to minimise the interference of urine phosphate with the colour development, and *cyclo*hexylsulphamic acid as a buffer system. The strip is dipped in the urine and the reaction is observed after 30 sec. and compared with a colour chart provided. A positive reaction is shown by a grey to blue colour, depending on the amount of phenylpyruvic acid present. The test is claimed to be more sensitive and specific than the liquid $FeCl_3$ test. H. F. W. KIRKPATRICK

1491. **α-Oxoglutaric acid and pyruvic acid in blood, cerebrospinal fluid and urine.** E. Zelnicek (Dept. Med. Chem., Masaryk Univ., Brno, Czechoslovakia). *Nature*, 1959, **184** (Suppl. No. 10), 727.—These keto acids are determined quantitatively after either chromatography or electrophoresis of their hydrazones (=0·5 ml of blood or urine, or 1 ml of c.s.f.). Electrophoresis is carried out in 0·05 *M* $NaHCO_3$ at 400 to 420 V and 10 to 18 mA for 5 hr. on Whatman No. 1 paper (20 cm × 29 cm). The chromatograms are developed in the solvent system *n*-butanol - ethanol - 1% aq. NH_3 (6:1:3). Electrophoresis does not separate the hydrazones of glyoxylic acid, phenylpyruvic acid and α-oxo*iso*caproic acid from the hydrazone of pyruvic acid. The separated hydrazone spots are extracted with *N* Na_2CO_3 and measured at 380 mμ. The average 24-hr. excretion of resting patients of α-oxoglutaric acid is 14·13 ± 3·20 mg and that of pyruvic acid is 8·16 ± 1·55 mg. Physical and mental strain increase these figures. S. BAAR

1492. **Simplified method for the determination of citric acid.** E. Beutler and M. K. Y. Yeh (Chicago Univ., Ill., U.S.A.). *J. Lab. Clin. Med.*, 1959, **54** (1), 125–131.—This is a modification of the pentabromoacetone method described by Natelson *et al.* (*J. Biol. Chem.*, 1948, **175**, 745) in which colour development is carried out with a thiourea - borax reagent. The method is suitable for biological fluids and has the advantage that special glass apparatus is not required. W. H. C. SHAW

1493. **Quantitative colorimetric method for estimation of 4-hydroxy-3-methoxymandelic acid in urine. Value in diagnosis of phaeochromocytoma.** M. Sandler and C. R. J. Ruthven (Bernhard Baron Mem. Res. Lab., Q. Charlotte's Matern. Hosp., London, England). *Lancet*, 1959, **ii**, 114–115.—The urine is passed through a cation-exchange resin (Dowex 1-X2 in acetate form). The adsorbed acid is eluted with *M* acetate buffer (pH 4·8), extracted with ethyl acetate and, after removal of the solvent, is autoclaved with 5% H_2SO_4 and alumina (catalyst) to convert it into vanillin. The vanillin is determined colorimetrically by reaction with indole in the presence of orthophosphoric acid and measurement at 495 mμ. The recovery averaged 90% with a reproducibility within ±10%. Some results for normal and pathological urines are given. H. F. W. KIRKPATRICK

1494. **Infra-red determination of gibberellins.** W. H. Washburn, F. A. Scheske and J. R. Schenck (Abbott Lab., N. Chicago, Ill., U.S.A.). *J. Agric. Food Chem.*, 1959, **7** (6), 420–422.—Gibberellic acid and gibberellin A, present together, are respectively determined by the measurement of the extinctions at 12·86 and 10·85 μ of a 15% soln. in pyridine. Results were in good agreement with those obtained by fluorimetric and radio-isotopic methods.
M. D. ANDERSON

1495. **Determination of gibberellins by derivative labelling with [^{14}C]diazomethane and by isotopic dilution analysis with tritium-labelled gibberellins.** W. E. Baumgartner, L. S. Lazer, A. M. Dalziel, E. V. Cardinal and E. L. Varner (Abbott Lab., N. Chicago, Ill., U.S.A.). *J. Agric. Food Chem.*, 1959, **7** (6), 422–425.—Gibberellic acid and gibberellin A are determined in fermentation broths by reaction with [^{14}C]diazomethane to yield the [^{14}C]methyl esters, followed by paper chromatography, examination with a Forro radiochromatogram scanner, and planimetric measurement of areas under the respective peaks. The sensitivity and accuracy can be increased at the expense of speed by extraction and liquid scintillation counting instead of scanning. The separation of gibberellins from process liquors is followed by adding tritium-labelled gibberellic acid, and assaying successive samples by liquid scintillation counting, with a single isolation to determine the activity of the final product. The methods are combined for the determination of gibberellins in plant tissues by double isotopic dilution analysis.
M. D. ANDERSON

1496. **Determination of bile acids in bile and duodenal contents by quantitative chromatography.** J. Sjövall (Dept. of Physiol. Chem., Univ. of Lund, Sweden). *Clin. Chim. Acta*, 1959, **4** (5), 652–664 (in English).—Direct paper chromatography of samples of bile and of duodenal contents is carried out with various phase systems according to the bile acid mixtures to be analysed.
H. F. W. KIRKPATRICK

1497. **Studies on the use of the Van den Bergh reagent for determination of serum bilirubin.** S. Meites and C. K. Hogg (Columbus Children's Hosp., Ohio, U.S.A.). *Clin. Chem.*, 1959, **5** (5), 470–478.—Modified reagents are proposed to increase the absorption of azobilirubin and to reduce the reaction time to less than 10 min. Diazo soln. *A* is prepared by dissolving sulphanilic acid (5 g) in conc. HCl (60 ml) and H_2O and diluting to 1 litre. Diazo soln. *B* is 2% $NaNO_2$ soln. For the blank soln., 60 ml of conc. HCl is diluted to 1 litre with H_2O. Haemolysis of the serum seriously affects the coupling, the azobilirubin produced apparently decreasing in direct proportion to the degree of haemolysis.
H. F. W. KIRKPATRICK

1498. **Spot tests for phospholipids and their use in paper chromatography.** G. J. M. Hooghwinkel, J. T. Hoogeveen, M. J. Lexmond and H. G. Bungenberg de Jong (Dept. Med. Chem., Univ. Leyden, Holland). *Proc. Kon. Akad. Wet., B*, 1959, **62** (4), 222–235 (in English).—Phosphatidylcholine, lysophosphatidylcholine, sphingomyelin, phosphatidylethanolamine and lysophosphatidylethanolamine were obtained by column chromatography from purified egg lecithin. These were separated on S. & S. paper No. 2043b by a modification of the method of Marinetti and Stotz (*Biochem. Biophys. Acta*, 1956, **21**, 168). The solvent used was di*iso*butyl ketone - acetic acid - H_2O (8 : 5 : 1). The papers were stained by (*i*) a "tricomplex" method (*cf.* Bungenberg de Jong and van Someren, *Proc. Acad. Sci. Amst.*, 1959) which yields red spots in the presence of phosphatides, (*ii*) a molybdic acid reagent, which hydrolyses the phosphatides so that they yield the molybdenum blue reaction, (*iii*) the dithiocarbamate (cephalin) reaction in which primary amines are converted by CS_2 into dithiocarbamate which yields a brown stain of Ag_2S with acid $AgNO_3$ soln., (*iv*) a Brilliant green - acid fuchsine reagent, which differentiates between the slightly and highly acidic substances, and (*v*) Rhodamine 6G (C.I. Basic Red 1), which reacts with various lipids and phospholipids to yield products that fluoresce in u.v. light. In addition, a molybdophosphoric acid reagent specific for choline and a ninhydrin reagent suitable for α-amino groups are described. The specificities of the various staining reagents are discussed.
S. BAAR

1499. **Serum phospholipid analysis by chromatography and infra-red spectrophotometry.** G. J. Nelson and N. K. Freeman (Donner Lab. of Biophysics, Univ. of California, Berkeley, U.S.A.). *J. Biol. Chem.*, 1959, **234** (6), 1375–1380.—A semi-micro method is described for which 10 ml of serum (equiv. to 15 to 30 mg of phospholipids) is required. The total lipids extracted with methanol - $CHCl_3$ are separated into five fractions by successive elution from a silicic acid - Celite column with dichloromethane, acetone, a mixture of 35% of methanol and 65% of dichloromethane (which elutes two fractions), and 95% methanol. The last three fractions contain the phospholipids and, by suitable i.r. measurements on these fractions dissolved in appropriate solvents, the amounts of lecithin, sphingomyelin, and a mixed fraction containing phosphatidylethanolamine and phosphatidylserine can be determined. The accuracy for a given component depends somewhat on the composition of the sample. In general, the error is $\pm 10\%$ for lecithin and sphingomyelin, and slightly greater for the mixed fraction.
J. N. ASHLEY

1500. **Micro-methods for the determination of chylomicron counts, fatty esters, lipid phosphorus and cholesterol in blood serum.** M. M. Clayton, P. A. Adams, G. B. Mahoney, S. W. Randall and E. Schwartz (Nutrition Res. Lab., Univ. of Maine, Orono, U.S.A.). *Clin. Chem.*, 1959, **5** (5), 426–444.—Published methods are adapted to micro-techniques.
H. F. W. KIRKPATRICK

1501. **Colour reactions for certain amino acids, amines and proteins.** R. F. Keeler (Vet. Res. Lab., Montana State Coll., Bozeman, U.S.A.). *Science*, 1959, **129**, 1618–1619.—Proteins, certain amino acids and amines yield pyrroles when allowed to react with acetonylacetone. The pyrroles couple with *p*-dimethylaminobenzaldehyde to yield coloured complexes. The specificity and possible uses of the reaction in colorimetric measurements and in the paper-chromatographic detection of these compounds are discussed.
H. F. W. KIRKPATRICK

1502. **Combined paper-electrophoretic and -chromatographic method for the determination of amino acids.** W. Matthias (Dtsch. Akad. d. Landwirtschaftwiss., Quedlinburg, Germany). *J. Chromatography*, 1959, **2** (4), 434–436 (in German).—A

method is described for the resolution of mixtures containing neutral, acid and basic amino acids. The mixture is applied to a narrow neck joining two unequal portions of a strip of chromatographic paper. The basic components are transferred by electrophoresis on to the short portion of the strip and cut off. A new piece is sewn on and the acid components are similarly transferred by reversing the electrodes. The basic fraction can now be resolved by chromatography on the long portion.

G. BURGER

1503. **Correction for the interference of urea in the determination of total α-amino acid nitrogen in plasma by the ninhydrin photometric method.** R. Rosenblum, M. Wolfman and L. Leiter (Montefiore Hosp., New York, U.S.A.). *J. Lab. Clin. Med.*, 1959, **54** (1), 132–135.—Parallel response lines for urea in the ninhydrin method are demonstrated in water, plasma and plasma filtrate. After the separate determination of urea in each plasma sample, a correction, based on the calibration in water, is applied to the total ninhydrin colour. Good agreement is attained with results by the manometric CO_2 method. W. H. C. SHAW

1504. **Micro-method for the polarographic determination of serine.** J. Ladik and I. Szekács (State Inst. of Hyg., Dept of Biochem., Budapest, Hungary). *Nature*, 1959, **184** (Suppl. No. 4), 188–189.—The method depends on the decrease in the height of the IO_4^- wave in the presence of serine. Sodium chloride soln. (0·85%) (2·8 ml), containing 0·01% of gelatin as max. suppressor, and 0·2 ml of 2×10^{-3} *M* KIO_4 are measured into each of two polarographic cells. To one is added 0·02 to 0·08 ml of aq. $1{\cdot}4 \times 10^{-3}$ *M* serine soln. and the soln. are allowed to stand at room temp. for 2·5 hr.; O is then removed in a stream of N, and the polarograms of the two soln. are recorded. The accuracy of the method is ±5 to ±10% depending on the concn. of serine. H. F. W. KIRKPATRICK

1505. **Micro-determination of ornithine.** I. Reifer and L. Buraczewska (Inst. Biochem. Biofiz. PAN, Warszawa). *Acta Biochim. Polon.*, 1959, **6** (2), 219–226.—Ornithine (**I**) is separated from other amino acids by paper chromatography with a solvent prepared by shaking *n*-butanol (100 ml) with 25% HCl (300 ml) and separating the alcoholic phase and mixing it with toluene (25 ml). The **I** is determined by the ninhydrin reaction. Lysine is not separated from **I** if present in concn. >25-fold.

B. LAKE

1506. **Quantitative determination of tryptophan metabolites (via kynurenine) in biological fluids.** D. Coppini, C. A. Benassi and M. Montorsi (Inst. of Pharm. Chem., Univ. of Padova, Italy). *Clin. Chem.*, 1959, **5** (5), 391–401.—A two-dimensional chromatographic method is described for the determination of the following substances in urine, blood or spinal fluid—kynurenine, 3-hydroxykynurenine, N-α-acetylkynurenine, anthranilic acid, 3-hydroxyanthranilic acid, kynurenic and xanthurenic acids, and the 8-methyl ether of xanthurenic acid. H. F. W. KIRKPATRICK

1507. **Determination of thyroxine levels in human plasma by double isotope-dilution technique.** J. K. Whitehead and D. Beale (Brit. Empire Cancer Campaign Radiochem. Lab., Middx. Hosp., London). *Clin. Chim. Acta*, 1959, **4** (5), 710–720.—In the method described, tritiated acetic anhydride is used as radioactive acetylating agent, with ^{131}I-labelled free thyroxine or ^{14}C-labelled N-acetylthyroxine as isotopic carrier. The method is accurate to within ±3%. The results of serum analyses are given.

H. F. W. KIRKPATRICK

1508. **Prevention of cross-contamination in the dry-ash method for protein-bound iodine.** J. K. Lugovoy (Chem. Dept., Bellevue Hosp., New York, U.S.A.). *Clin. Chem.*, 1959, **5** (5), 445.—Tubes are placed in the muffle-furnace with the open ends slightly protruding and the contents are allowed to char with the furnace door open. When no more smoke is emitted the tubes are pushed completely into the furnace, the door is closed and the incineration completed. H. F. W. KIRKPATRICK

1509. **Physical and chemical studies of a limited reaction of iodine with proteins. [Determination of mercapto groups.]** L. W. Cunningham and B. J. Nuenke (Dept. of Biochem., Vanderbilt Univ. Sch. of Med., Nashville, Tenn., U.S.A.). *J. Biol. Chem.*, 1959, **234** (6), 1447–1451.—At low temp. and nearly neutral pH the mercapto groups of proteins react with iodine in KI soln. to form protein sulphenyl iodide, and a spectrophotometric method of determination of mercapto groups is based on the extinction of the tri-iodide ion at 355 mμ. A soln. of the protein in phosphate buffer (pH 6·5), 0·2 *M* in KI, at 0° to 5° is titrated with 0·001 *N* iodine in 0·2 *M* KI until the extinction becomes constant, or until its decrease becomes small and linear.

J. N. ASHLEY

1510. **Far-ultra-violet direct photometry applied to protein electrophoresis.** N. Ressler and S. D. Jacobson (Wayne County Gen. Hosp., Eloise, Mich., U.S.A.). *J. Lab. Clin. Med.*, 1959, **54** (1), 115–124.—The electrophoresis of serum proteins is carried out in tri(hydroxymethyl)methylamine buffer supported by a weak agar gel on a horizontal quartz plate. After separation, photographs are taken through the buffer with u.v. radiation (200 mμ) obtained from a monochromator, and the proteins are determined from densitometer readings on the developed photograph. Similar absorption coefficients are given by the various protein fractions examined, and Beer's law is obeyed. Good agreement was attained with results by conventional staining techniques. The factors affecting resolution are discussed. W. H. C. SHAW

1511. **Determining serum proteins gravimetrically after agar electrophoresis.** R. D. Strickland, P. A. Mack, F. T. Gurule, T. R. Podleski, O. Salome and W. A. Childs (Res. Div., Vet. Admin. Hosp., Albuquerque, N.M., U.S.A.). *Anal. Chem.*, 1959, **31** (8), 1410–1413.—The fractions are visualised by the production of turbid areas when the agar is immersed in a bath of 10% trichloroacetic acid soln. These areas are cut out and removed to 15-ml centrifuge tubes, 2 ml of H_2O is added and the mixture is heated to melt the agar; 2 ml of 20% trichloroacetic acid soln. is added and the ppt. is centrifuged, transferred to a micro-crucible (described) and weighed.

H. F. W. KIRKPATRICK

1512. **Quantitation of plasma proteins on cellulose acetate strips.** F. Albert-Recht (Dept. of Clin. Chem., Univ. of Edinburgh, Scotland). *Clin. Chim. Acta*, 1959, **4** (5), 627–638.—Apparatus for electrophoresis and scanning and calculations of correction factors are described for the method of Kohn (*Anal. Abstr.*, 1958, **5**, 1747).

H. F. W. KIRKPATRICK

1513. **Dye-binding capacities of eleven electrophoretically separated serum proteins.** R. D. Strickland, T. R. Podleski, F. T. Gurule, M. T. Freeman and W. A. Childs (Res. Div., Vet. Admin. Hosp., Albuquerque, N.M., U.S.A.). *Anal. Chem.*, 1959, **31** (8), 1408–1410.—Isolated fractions obtained by agar electrophoresis were used in tests with bromophenol blue, Amido black 10B (C.I. Acid Black 1) and Ponceau 2R (C.I. Acid Red 26). Wide variations were observed among the different fractions from the same serum, and also among the corresponding fractions from different sera. It is concluded that only gross abnormalities in protein distribution are detectable by staining methods.

H. F. W. KIRKPATRICK

1514. **New method for the quantitative determination of haptoglobin.** F. A. Hommes (Dept. of Physiol. Chem., Univ. of Nijmegen, The Netherlands). *Clin. Chim. Acta*, 1959, **4** (5), 707–709 (in English).—An excess of carboxyhaemoglobin is added to serum and the complex is separated from the free haemoglobin by electrophoresis with 0·05 *M* phosphate buffer (pH 6·9). Free haemoglobin migrates to the anode and the complex to the cathode. The band due to the complex is eluted and the peroxidase activity is determined by a slight modification of the method of Chance and Maehly ("Methods in Enzymology," Vol. II, New York, 1955, p. 764). H. F. W. KIRKPATRICK

1515. **Discontinuous buffer system for paper electrophoresis of human haemoglobins.** C. A. J. Goldberg (William Pepper Lab. of Clin. Med., Univ. of Pennsylvania, Philadelphia, U.S.A.). *Clin. Chem.*, 1959, **5** (5), 446–451.—The electrode vessels are filled with the usual barbitone buffer soln. (pH 8·6), and the paper is immersed in a buffer soln. (pH 9·1) prepared by dissolving tri(hydroxymethyl)methylamine (6·05 g), disodium EDTA dihydrate (0·78 g) and boric acid (0·46 g) in H_2O and making up the vol. to 500 ml. The paper is blotted very lightly and placed in the electrophoresis chamber, buffered haemoglobin soln. (*Ibid.*, 1957, **3**, 1) is applied and the process is carried out in the usual way. The resolution of abnormal haemoglobins is superior to that in either buffer soln. alone, and enables haemoglobin A_2 to be detected in small samples of haemolysate. The staining, with Amido black 10B (C.I. Acid Black 1), of patterns that on visual inspection show only haemoglobin A, reveals the A_2 component if present.

H. F. W. KIRKPATRICK

1516. **Routine micro-estimation of iron in haemoglobin.** J. Fischl (Chem. Lab., "Assaf Harofe" Govt. Hosp., Zrifin, Israel). *Clin. Chim. Acta*, 1959, **4** (5), 686–690.—The proposed method combines simplicity with good precision. *Procedure*—Pipette 0·02 ml of blood into 5 drops of H_2O contained in a 15-ml centrifuge tube, add 0·3 ml of conc. H_2SO_4 and 0·5 ml of saturated aq. potassium persulphate soln. Cool and add 5·0 ml of colour reagent (to 30 g of KSCN dissolved in 100 ml of H_2O add 4 ml of acetone and 500 ml of *iso*butyl alcohol), shake vigorously and centrifuge at high speed for 10 min. Measure the colour of the supernatant soln., and also that of a standard (10 µg of Fe in 0·1 ml) and a blank prepared similarly, against *iso*butyl alcohol.

H. F. W. KIRKPATRICK

1517. **Practical method for the determination of fibrinogen.** M. Reiner and H. L. Cheung (Dept. of Lab., D.C. Gen. Hosp., Washington, U.S.A.). *Clin. Chem.*, 1959, **5** (5), 414–420.—In the proposed method, fibrinogen is converted by thrombin into fibrin. The clot is dissolved in hot NaOH soln. and the protein is determined by the biuret or the more sensitive phenol - biuret method.

H. F. W. KIRKPATRICK

1518. **Ultracentrifugal method for the determination of serum lipoproteins.** L. Del Gatto, F. T. Lindgren and A. V. Nichols (Donner Lab., Dept. of Phys., Univ. of Calif., Berkeley, U.S.A.). *Anal. Chem.*, 1959, **31** (8), 1397–1399.—The isolation and analysis of lipoproteins in a NaBr medium, of density 1·20 g per ml, is described.

H. F. W. KIRKPATRICK

1519. **The separation of lipo- and glyco-proteins of serum by paper electrophoresis.** A. Rovescalli (Lab. Med. Prov., Milano, Italy). *Attual. Lab.*, 1959, **5** (4), 97–104.—A new buffer soln. at pH 8·6 is proposed (barbitone sodium, 5·88 g; Na acetate, 3·88 g; K oxalate, 1·38 g; and 0·1 *N* HCl, 200 ml; made up to 1 litre). Dialysis against 20% poly(vinylpyrrolidone) soln. is also suggested to concentrate the serum and avoid successive additions of serum to the spot as in normal electrophoresis. It is proposed to pre-stain the serum before electrophoresis by the following procedure. The serum (0·2 ml) is treated with 2 drops of stain [2 to 3 g of Sudan Black B (C.I. Solvent Black 3) dissolved by heating in 100 ml of propane-1 : 2-diol and filtered]. The mixture is shaken slowly and maintained at 37° for 1 hr., and then centrifuged, and the supernatant liquid is submitted to electrophoresis.

L. ZANONI

1520. **Analysis of adenine polyphosphates by paper chromatography.** G. P. Briner (Dept. of Pharmacol., Univ. Melbourne, Australia). *J. Chromatography*, 1959, **2** (4), 418–422 (in English).—A method is described for the extraction of adenine mono-, di- and tri-phosphates from frog muscle, their separation by paper chromatography and their determination by spectrophotometry at 260 mµ. An analysis for further components can be made on the same extract. A complete assay of 8 muscles can be made in 24 hr. The procedure described must be strictly adhered to or the reproducibility is poor. G. BURGER

1521. **Studies of cholesterol methodology and application to population surveys.** N. F. Hollinger, E. Austin, D. Chandler and R. K. Lansing (Sch. of Public Health, Univ. of Calif., Berkeley, U.S.A.). *Clin. Chem.*, 1959, **5** (5), 458–464.—A comparison is made of the results of replicate analyses by seven different methods. On the basis of reproducibility and ease of performance the method of Carr and Drekter (*Anal. Abstr.*, 1957, **4**, 1945) is concluded to be the most suitable.

H. F. W. KIRKPATRICK

1522. **Determination of blood cholesterol by colorimetry of the digitonide. Operating improvements, particularly in the case of high hyperlipaemias.** M. Herbain (Serv. de Rech., Roussel-Uclaf, Romainville, Seine, France). *Bull. Soc. Chim. Biol.*, 1959, **41** (5–6), 821–833.—The method of Sperry and Webb (*J. Biol. Chem.*, 1950, **187**, 97) for cholesterol (**I**) is modified to enable whole blood to be used even in cases of hyperlipaemia. The instantaneous

mixing of the blood with the extracting solvent is effected by pipetting the blood (0·1 ml) into a 1-ml pocket in the side of a special 20-ml centrifuge tube (illustrated) and haemolysing it by mixing it in the pocket with 0·1 ml of water. The solvent (ethanol - acetone) (1:1) (5 ml) is placed in the rest of the tube and the stoppered tube is inverted and shaken. The tube is centrifuged for 5 min. at 3000 r.p.m. without heating. Free **I** is determined directly on 2 ml of the clear supernatant extract by pptn. with digitonin and application to the ppt. of the Pesez reaction (*cf. Anal. Abstr.*, 1960, **7**, 1524), with a Meunier photometer and No. 49 filter. Total **I** is determined in the same way on 1 ml of the extract after hydrolysis with KOH. The modifications are equally applicable to serum or plasma.

E. J. H. BIRCH

1523. **Fractionation of cholesteryl esters by silicic acid chromatography.** P. D. Klein and E. T. Janssen (Div. of Biol. and Med. Res., Argonne Nat. Lab., Lemont, Ill., U.S.A.). *J. Biol. Chem.*, 1959, **234** (6), 1417–1420.—A method is described for the separation of mixtures of cholesteryl esters into four fractions (saturated, oleate, linoleate and arachidonate esters) on a silica column conditioned by pre-treatment with 5% of benzene in hexane. The esters, dissolved in hexane containing 13% of benzene, are applied to the column and are eluted with the same solvent mixture. The method compares favourably with other methods that involve hydrolysis of the esters, and there is the added advantage that the esters are isolated as individual compounds, and are not destroyed.

J. N. ASHLEY

1524. **Halochromy and halofluorescence of 19-norsteroids and aetiocalciferols.** M. Pesez. *Bull. Soc. Chim. France*, 1958, **40**, 369–371.—A series of 3-hydroxylated steroids was submitted to the following reaction. A dil. soln. of steroid in acetic acid (0·2 ml) was treated with a reagent prepared by diluting a 5% HgO soln. in 20% H_2SO_4 (2 ml) to 10 ml with cold H_2SO_4 - acetic acid (2:1). The colours obtained were compared with those given by the Liebermann - Burchard and Denigès reactions.

N. E.

1525. **Elimination of interfering chromogens in the Zimmermann reaction for measuring 17-oxosteroids.** A. G. Ware, J. A. Demetriou, S. Notrica, R. Searcy, C. Walberg and F. Cox (Los Angeles Gen. Hosp., Calif., U.S.A.). *Clin. Chem.*, 1959, **5** (5), 479–487.—Colour development is carried out by the usual procedure, then 40% aq. *n*-propanol (6 ml) and dichloromethane (3 ml) are added in that order. The tubes are shaken vigorously and centrifuged for a few minutes at 2000 r.p.m. The colour in the lower layer is measured at 540 mμ, after removal of the upper aq. layer containing the brown chromogens. No correction of the readings is necessary.

H. F. W. KIRKPATRICK

1526. **Determination of cortisol and cortisone in urine by means of acetic anhydride labelled with tritium.** E. Demey (Lab. de Clin. Méd. et de Rech. Chim., Univ. de Liége, Belgium). *Bull. Soc. Chim. Biol.*, 1959, **41** (5–6), 795–803.—*Procedure*—Urine (100 ml) is treated with a 1% soln. of *Helix pomatia* digestive juice, and then adjusted to pH 5 and incubated for 18 hr. at 37°. The urine is then extracted with dichloromethane at once and again after 24 hr. at 37°. After being washed with 0·01 *N* NaOH and water the combined extracts are filtered through anhyd. Na_2SO_4 and evaporated *in vacuo*. The residue is acetylated with labelled acetic anhydride and pyridine in a sealed system, and known quantities (10 mg) of the unlabelled acetates of cortisol and cortisone are then added. The mixture (0·5 mg) is placed on the starting line of a strip of Whatman No. 1 paper impregnated with formamide, and chromatographed with hexane satd. with formamide for 16 hr. at 22°. The acetates remain on the starting line and are then chromatographed with benzene satd. with formamide for 12 hr. at 22°. The chromatograms are dried at room temp. *in vacuo*, laid on sheets of photographic paper, and exposed to u.v. light which is absorbed by the steroids; the position of the compounds is thus indicated and the cortisol (lower R_F) and cortisone spots are cut out and eluted between strips of metal with 20 to 30 ml of ethanol. The soln. are then evaporated *in vacuo*. The cortisol acetate is further purified by chromatography on paper impregnated with propane-1:2-diol (**I**) (30% in acetone), with toluene satd. with **I** as mobile phase, for 20 hr. at 22°. The cortisone acetate is similarly purified but on paper impregnated with 40% of **I** in acetone. The acetates are detected and extracted from the paper as before. The activity of each compound is determined by using a counter (Tracerlab SC 16) and the total amount of each is determined by u.v. spectrophotometry at 240 mμ. From a knowledge of the original specific activity of the acetic anhydride the quantities in the original urine can then be calculated. An accuracy of ±5% is attained.

E. J. H. BIRCH

1527. **Variation of pH of clinical samples as a source of error in enzyme determinations.** R. J. Henry and N. Chiamori (Bio-Science Res. Found., 12318 Santa Monica Blvd., Los Angeles, Calif., U.S.A.). *Clin. Chem.*, 1959, **5** (5), 402–404.—Buffer soln. employed to control pH may be inadequate to cope with the variations in pH of urine or serum samples occurring physiologically or as a result of change on standing. Errors arising from this source are demonstrated in an investigation of the alkaline phosphatase method of Shinowara *et al.* (*J. Biol. Chem.*, 1942, **142**, 921).

H. F. W. KIRKPATRICK

1528. **Determination of the urease activity of soya meal.** C. van Rede (Lab. d. N.V. Mij. tot Exploit. Vereenigde Oliefab., Zwijndrecht, Netherlands). *Chem. Weekbl.*, 1959, **55** (25), 294–295.—The method of Dangoumau and Debruyne (*Rev. Corps Gras*, 1955, **2**, 390) is simplified. *Procedure*—To 2 g of the ground sample in a 100-ml flask add 10 ml of buffer (pH 6·9) (1·0 g of $NaH_2PO_4.H_2O$ and 4·0 g of $Na_2HPO_4.12H_2O$ per 100 ml) and 10 ml of 10% urea soln., both at 20°, and maintain the flask at 20° for exactly 5 min., swirling at intervals. Add ≃0·2 g of MgO, make up to 100 ml with water and mix well. Transfer the mixture to a Kjeldahl flask containing 250 ml of previously boiled water and a few mg of an anti-foaming agent. Heat to boiling-point within 5 min. and distil off ≃100 ml in 25 min. into 25 or 50 ml of 0·1 *N* H_2SO_4 (according to the urease content). Titrate with 0·1 *N* NaOH, with a Tashiro indicator. Carry out a blank simultaneously. For urease contents >30 units per g the sample size should be decreased.

M. J. MAURICE

1529. **Serum quinine oxidase. A specific test for parenchymatous liver damage.** W. Roman and A. Dulmanis (Inst. Med. Vet. Sci., Adelaide, S. Australia). *Lancet*, 1959, **ii**, 90.—*Procedure*—Mix 1 ml of serum with 3 ml of 0·06 *M* phosphate buffer soln. (pH 7·2), add 1 ml of quinine hydrochloride soln. (25 mg per 100 ml in H_2O) and measure the extinction in a 1-cm cell at 366 mμ exactly 1 min. after mixing. After 10 min. at 20° repeat the reading. An increase of 0·001 in the extinction represents 1 unit of enzyme activity. In normal sera (60), myocardial infarction (20), pancreatitis (7) and biliary obstruction (20), less than 5 units were found. In cases of infective hepatitis (48), the values ranged from 7 to 89 units. Out of a total of 20 cases of cirrhosis, raised values were found in 3 cases with continuing liver-tissue destruction, the other values being <5 units. H. F. W. Kirkpatrick

1530. **Studies on choline dehydrogenase. I. Extraction in soluble form, assay, and some properties of the enzyme.** G. Rendina and T. P. Singer (Edsel B. Ford Inst. for Med. Res., Henry Ford Hosp., Detroit, Mich., U.S.A.). *J. Biol. Chem.*, 1959, **234** (6), 1605–1610.—Choline dehydrogenase activity is determined in mitochondrial as well as in soluble preparations with phenazine methosulphate as the electron acceptor. Other acceptors usually employed for this enzyme (*e.g.*, cytochrome *c*, ferricyanide, methylene blue and 2:6-dichlorophenolindophenol) either do not react directly with the dehydrogenase and thus measure only a part of the activity, or they are unreliable because of the extensive dependence of the measured activity on dye concn. Assays are carried out by the manometric method (*cf.* Singer and Kearney, "Methods of Biochemical Analysis," Interscience Publishers Inc., New York, 1957, Vol. IV, p. 307) in the presence of 5×10^{-3} *M* phosphate (pH 7·6), 10^{-3} *M* cyanide, $3·3 \times 10^{-2}$ *M* choline, and 0·5 mg of phenazine methosulphate per ml. All the components except the acceptor are in the main compartment of the Warburg vessel. After suitable temp. equilibration, the contents of the side arm are tipped in and manometric changes are recorded for the period from 2 to 7 min. after mixing. J. N. Ashley

1531. **The enzymic reduction of hydroxyguanidine. [Determination of hydroxyguanidine reductase.]** J. B. Walker and M. S. Walker (Dept. of Biochem., Baylor Univ. Coll. of Med., Houston, Tex., U.S.A.). *J. Biol. Chem.*, 1959, **234** (6), 1481–1484.—An assay for the reductase is described in which reduced riboflavine phosphate is used as the electron donor. The enzyme suspension is incubated with riboflavine phosphate, hydroxyguanidine, $Na_2S_2O_4$ and phosphate buffer (pH 7·4) at 25° for 30 min. The reaction is stopped by the addition of trichloroacetic acid, and after centrifuging, the supernatant soln. is assayed for guanidine by using the diacetyl method (Eggleton *et al.*, *Brit. Abstr. C*, 1944, 29) or for the remaining hydroxyguanidine by using the sodium pentacyanoamminoferrate method (Walker, *Anal. Abstr.*, 1958, **5**, 4243). Hydroxyguanidine interferes somewhat with the diacetyl method, but guanidine does not affect the hydroxyguanidine assay. J. N. Ashley

1532. **Colorimetric determination of glutamic-oxalacetic transaminase in serum or plasma.** D. F. Ashman (Dept. de Bioquím., Univ. del Zulia, Maracaibo). *Acta Cient. Venezolana*, 1959, **10** (1), 14–15.—The method depends on the colorimetric determination of the pyruvic acid produced in the reaction between DL-aspartic acid and α-oxoglutaric acid, and is a modification of one described earlier (*cf.* Cabaud *et al.*, *Anal. Abstr.*, 1957, **4**, 2352). C. A. Blau

1533. **The determination of transaminases by photometric methods with 2:4-dinitrophenylhydrazine.** F. Candura (Ist. di Med. del Lavoro, Univ. di Pavia, Italy). *Boll. Soc. Ital. Biol. Sper.*, 1959, **35** (12), 697–700.—In photometric determinations of glutamate-pyruvate transaminase activity, the extinction value of the sample was sometimes less than that of the appropriate blank. Mitochondrial preparations from rat and hen liver were allowed to act on buffered digests (pH 7·45) containing 2 micromoles of pyruvate, or 200 micromoles of alanine plus 2 micromoles of α-oxoglutarate. When the resulting keto acids were determined as 2:4-dinitrophenylhydrazones, the extinction value obtained for the pyruvate system was less than that of the control (reaction stopped at zero time) by an amount corresponding to the increase in extinction value obtained for the alanine-oxoglutarate system. Transamination thus appeared to be masked, wholly or in part, by metabolisation of pyruvate through another route. Three approaches to the solution of the problem of the evaluation of the glutamate-pyruvate system are suggested—(*i*) determination of the amino acids rather than the keto acids; (*ii*) the immediate reduction of the pyruvate formed by the addition of an excess of lactic dehydrogenase and reduced diphosphopyridine nucleotide, and spectrophotometric measurement of the diphosphopyridine nucleotide produced; and (*iii*) the separate determination of the capacity of the test system to metabolise pyruvate alone, so that a suitable correction can be applied to the transaminase system. C. Rainbow

1534. **Regulation of activity of phosphoribosyl pyrophosphate amidotransferase by purine nucleotides: potential feedback control of purine biosynthesis. [Enzyme assay.]** J. B. Wyngaarden and D. M. Ashton (Dept. of Med., Duke Univ. Sch. of Med., Durham, N.C., U.S.A.). *J. Biol. Chem.*, 1959, **234** (6), 1492–1496.—The assay is based on the measurement of the rate of formation of glutamic acid (**I**). The amidotransferase catalyses the formation of phosphoribosylamine from phosphoribosyl pyrophosphate and glutamine, which is deamidated to **I**. The activity of the transferase is determined by combining this reaction with that for the determination of **I** using glutamic dehydrogenase and the 3-acetylpyridine analogue of diphosphopyridine nucleotide as H acceptor. The oxidation of **I** goes to completion and the reduction of the analogue is followed spectrophotometrically at 363 mμ. J. N. Ashley

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See also Abstracts—**1258**, Autoradiography of tritium-labelled compounds. **1295**, Boron in blood-ash. **1352**, Iron in culture media. **1466**, Hydroxyproline as a measure of collagen. **1561**, Glutamic acid in molasses, etc. **1605**, Chromatography of guanidine derivatives.

Pharmaceutical analysis

1535. **Contribution to the microscopic characterisation and identification of drugs. I.** M. Brandstätter-Kuhnert and A. Kofler (Pharmakogn. Inst., Univ.,

Innsbruck). *Sci. Pharm.*, 1959, **27** (2), 73–81.—Melting-points, eutectic temperatures, refractive indices and other characteristic properties of 40 drugs are tabulated. A. R. ROGERS

1536. **Adaptation of Mayer's reagent as a spray reagent for detecting alkaloids on paper chromatograms.** S. C. Pan and G. H. Wagman (Squibb Inst. for Med. Res., New Brunswick, N.J., U.S.A.). *J. Chromatography*, 1959, **2** (4), 428 (in English).—Alkaloid spots on paper chromatograms can be located by spraying the paper with a 1% aq. soln. of $HgCl_2$, drying and then spraying with a 0·5% aq. soln. of KI. The spots appear white on a salmon background. The method is not specific and does not identify alkaloids. G. BURGER

1537. **Polarographic determination of ephedrine.** G. Dušinský and T. Čavaňák (State Inst. for Control of Drugs, Bratislava, Czechoslovakia). *Českosl. Farm.*, 1959, **8** (5), 205–208.—When treated with bromine, ephedrine is converted into polarographically active benzaldehyde. *Procedure*—Dissolve the sample in a phosphate buffer soln. of pH 6·5 to 7·0. To 1 ml of this soln. containing 0·25 to 1 mg of ephedrine add 2 *N* KCl (1 ml) and dilute to 3 ml with phosphate buffer soln. Add a satd. soln. of bromine in H_2O (1 ml) and heat the stoppered tube in a boiling-water bath for 2 min. Cool, add methanol (2·5 ml) and phosphate buffer soln. (3·5 ml). Remove O with a stream of N and register the polarographic wave from −0·8 V. Compare with a calibration curve. The method was tested and gave good results for the analysis of pharmaceutical products, *e.g.*, tablets and injections, and is suitable for the determination of ephedrine in the presence of pentamethylenetetrazole, acetylsalicylic acid, NNN′N′-tetraethylphthalamide, phenylmercury borate, NaCl, Na_2SO_4, lactose, sucrose and starch. J. ZÝKA

1538. **Qualitative and quantitative assay of tropane alkaloids in drugs and pharmaceutical preparations by means of paper chromatography.** W. Dębska and K. Kostujak (Dept. of Anal. and Technol. of Herbal Medicines, Poznań, Poland). *Biul. Inst. Roślin Leczniczych*, 1959, **5** (2), 97–106.—The method deals chiefly with the separation and determination of atropine and hyoscine. For detection, 0·5 g of finely powdered sample is shaken with 10 ml of benzene and 1 ml of 10% aq. NH_3, then filtered, and 0·2 to 0·4 ml of filtrate (±0·01 ml) is transferred on to strips of Whatman paper No. 3 (15 cm × 57 cm) and chromatographed by the descending technique with water-saturated *n*-butanol - glacial acetic acid (25 : 1) for about 18 hr. The chromatogram is dried and sprayed with Dragendorff's reagent. Atropine (R_F 0·53) moves faster than hyoscine (R_F 0·35). For quant. determinations, Dragendorff's reagent is applied only to a chromatogram prepared from a standard soln. Corresponding areas on the chromatogram of the sample are cut out, eluted with ethanol and determined colorimetrically. The separation of alkaloids from impurities was equally satisfactory when diethyl ether, $CHCl_3$ or ethanol was used instead of benzene. W. B. MIASKOWSKI

1539. **Isolation and detection of ergometrine in toxicological analysis.** A. S. Curry (Home Office Forensic Sci. Lab., Harrogate, England). *J. Pharm. Pharmacol.*, 1959, **11** (7), 411–414.—Macerate the sample of tissue (*e.g.*, buttock, 60 to 200 g) with 5% acetic acid soln. (350 ml) in the presence of sufficient $(NH_4)_2SO_4$ to saturate the soln. Heat in a bath of boiling water for 30 min., filter through a pad of paper pulp and wash the tissue on the pad with hot 5% acetic acid soln. (2 × 250 ml). Cool the combined filtrate and washings, extract with an equal volume of diethyl ether and reject the ether extract. Add an excess of aq. NH_3 to the aq. phase, saturate with $(NH_4)_2SO_4$ and extract with two equal volumes of diethyl ether. Samples of urine may be extracted in the same way after the addition of $(NH_4)_2SO_4$. The recovery of ergometrine at this stage is ≃40%. Evaporate the ether and submit the crude alkaloidal residue to chromatography on paper buffered with 5% sodium dihydrogen citrate with the *n*-butanol - citric acid solvent system of Curry and Powell (*Nature*, 1954, **173**, 1143). To detect the ergometrine, at R_F 0·23, observe the fluorescence in u.v. light or spray the paper with a soln. of 0·5% *p*-dimethylaminobenzaldehyde (**I**) and 10% H_2SO_4 in ethanol, heat gently and observe the blue - violet colour. As little as 0·1 µg of ergometrine can be detected. None of the constituents found in a normal buttock tissue causes interference. Procaine, with R_F = 0·25, gives a yellow colour with **I**. A. R. ROGERS

1540. **Rapid spectrophotometric determination of total nicotine alkaloids in tobacco smoke.** S. W. Pleasants, C. M. Haynes and L. S. Harrow (Res. and Devel. Dept., Philip Morris Inc., Richmond, Va., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1959, **42** (2), 424–427.—The method of Willits *et al.* (*Anal. Chem.*, 1950, **22**, 430) as modified by Griffith (*Tobacco Sci.*, 1957, **1**, 130) has been adapted to the determination of nicotine alkaloids in tobacco smoke. An aq. soln. of the smoke particulate phase is distilled with steam, first in the presence of HCl and then after rendering the residue alkaline. The latter distillate is collected in 3 *N* H_2SO_4 and examined spectrophotometrically at 260 mµ. The method is more specific than the $HClO_4$ method of Cundiff and Markunas (*cf. Anal. Abstr.*, 1956, **3**, 814), and the results are more precise. A. A. ELDRIDGE

1541. **Assay of caffeine and theobromine in an anhydrous medium with perchloric acid solution.** M. Rink and R. Lux (Pharm. Inst., Univ., Bonn). *Dtsch. ApothZtg*, 1959, **99** (41), 1051–1054.—*Procedure*—To determine caffeine, dissolve the sample (0·2 g) in benzene (20 ml) with warming, then cool, add 2 or 3 drops of indicator soln. and titrate with 0·1 *N* $HClO_4$ in glacial acetic acid. Suitable indicators are Sudan III (C.I. Solvent Red 23) (**I**), Sudan IV (C.I. Solvent Red 4) (0·5% soln. in $CHCl_3$), Nile blue A (C.I. Basic Blue 12) (**II**), neutral red, malachite green (C.I. Basic Green 4) (**III**) or crystal violet (0·5% soln. in glacial acetic acid) or brilliant cresyl blue (C.I. 51010) (0·25% soln. in $CHCl_3$). To determine theobromine, dissolve the sample (0·2 g) in glacial acetic acid (5 ml) with warming, add hot benzene (25 ml) and titrate hot with 0·1 *N* $HClO_4$ in acetic acid, with **I**, **II** or **III** as indicator. A. G. COOPER

1542. **Contribution to the chemical evaluation of digitalis drugs (*D. purpurea* and *D. lanata*). II.** W. Hauser and W. Berger (Pharmakogn. Inst., Univ., Graz). *Sci. Pharm.*, 1959, **27** (2), 97–100.—A loss of glycosides occurs in the procedure given previously (*cf.* Hauser and Mathauser, *Ibid.*, 1958, **26**, 258) for the determination of genins

following lead acetate purification of a hot-water extract of the drug. The loss is proportionately greater, the higher the glycoside content of the drug, and it amounts to about 25% with *D. lanata*.

A. R. ROGERS

1543. **Quantitative determination of cyanocobalamin.** H. Cords and O. T. Ratycz (Squibb Inst. for Med. Res., New Brunswick, N.J.). *Drug Standards*, 1959, **27** (5), 132–136.—The method is suitable for the determination of cyanocobalamin (**I**) in solid and liquid preparations of vitamin B_{12} containing <70% of impurities, in which >70% of the total vitamin B_{12} (**II**) content is in the form of **I**. *Procedure*—Mix an aliquot of the sample with a 0·1% soln. of KCN in aq. acetic acid of pH 6 and determine **II** by measurement of the extinction at 550 mμ. To determine the ratio of **I** to **II**, apply another sample (≃1 mg) to Whatman No. 3MM paper and develop for 18 hr. in subdued light by the descending technique with H_2O-satd. *sec.*-butyl alcohol containing 1% of glacial acetic acid; dry the chromatogram at room temp., cut out the zones of **I** and of other cobalamins, separately elute with KCN - acetic acid soln. and measure at 550 mμ. Calculate the content of **I** by correction of **II** by the ratio of **I** to **II**.

A. R. ROGERS

1544. **Micro-determination of cynarin (1:4-dicaffeylquinic acid).** G. Ghielmetti and M. Bacchini (Lab. Ric. Fitochim. "Inverni & Della Beffa," Milano, Italy). *Farmaco, Ed. Prat.*, 1959, **14** (5), 284–288.—*Procedure*—A preliminary chromatographic separation of cynarin (**I**) (40 to 80 μg in ethanol) is carried out on Whatman No. 1 paper, with methanol - benzene - *iso*amyl alcohol - H_2O (2:1:1:1), by ascending chromatography on two sheets of paper. On one of these **I** is identified with ammoniacal $AgNO_3$ soln., and on the other paper the spot corresponding to **I** is cut out and eluted with 50% methanol, the eluate being collected in a 10-ml flask. The absorption maximum is at 322 mμ. The mean error is 3 to 4%. The determination can also be made by measuring the area of the spot. In the range from 20 μg to 80 μg the area in sq. cm is proportional to the amount of **I**. The mean error by this method is 7 to 8%.

L. ZANONI

1545. **Comparative studies on the metabolism, excretion and identification of barbiturate-type hypnotics.** H.-H. Frey, F. Sudendey and D. Krause (Pharmacol. Inst., Veterinary Coll., Hanover). *Arzneimittel-Forsch.*, 1959, **9** (5), 294–297.—Barbitone, butabarbital, cyclobarbitone, hexobarbitone, pentobarbitone, phenobarbitone and propallylonal and their metabolites in urine were detected by means of ascending paper chromatography. The separation was effected with a *n*-butanol - $CHCl_3$ - aq. NH_3 solvent system and the spots were located with a satd. soln. of $HgNO_3$. The lower limits of detection were 1 to 2 μg (hexobarbitone, 10 μg). The nature of the metabolites detected, and the amount excreted unchanged, were characteristic for each drug, thus permitting identification of the barbiturate administered.

A. G. COOPER

1546. **Quantitative determination of drugs in mixtures and galenicals. I. Amidopyrine.** N. Kreuzkamp, S. Ebel and H. Kindling (Pharm.-chem. Inst. d. Univ., Marburg/Lahn). *Dtsch. ApothZtg*, 1959, **99** (19), 433–436.—Amidopyrine (**I**) may be titrated in the presence of several neutral substances with 0·5 *N* H_2SO_4, with methyl orange as indicator. In slightly alkaline medium **I** can be determined by oxidation to N-acetyl-N-methyl-N′-dimethyloxamoylphenylhydrazine with NH_4VO_3 or $KMnO_4$. For the acidimetric determination, 200 mg of **I** is needed, for the oxidimetric method only 30 to 40 mg. Other methods tested were (*i*) back-titration of an excess of picric acid and (*ii*) complexometric determination of a **I** - $Cd(SCN)_2$ complex. These are more complicated and cannot be used in the presence of such a variety of compounds as the acidimetric and oxidimetric methods.

M. H. SAWISTOWSKA

1547. **Identification of phenazocine [2′-hydroxy-5:9-dimethyl-2-phenethyl-6:7-benzomorphan], a potent new analgesic.** E. G. C. Clarke (Dept. of Physiology, Royal Veterinary College, London). *Nature*, 1959, **184** (Suppl. No. 7), 451.—Phenazocine (**I**) may be identified by colour and crystal tests described previously (*cf.* Clarke and Williams, *J. Pharm. Pharmacol.*, 1955, **7**, 255; Clarke, *Ibid.*, 1958, **10**, 194). Crystal tests with KI soln., Na_2CO_3 soln. and picrolonic acid soln. may be used to distinguish the (−)-isomer of **I** from the racemic mixture and also from the analgesic compound 2′-hydroxy-2:5:9-trimethyl-6:7-benzomorphan (**II**). **I** and **II** may be separated by paper chromatography with the *n*-butanol - citric acid solvent of Curry and Powell (*cf. Nature*, 1954, **173**, 1143).

A. R. ROGERS

1548. **New method for the detection and rapid quantitative determination of amidopyrine in the presence of commonly accompanying substances.** J. Dick. *Rev. Chim., Acad. R.P.R.*, 1959, **4** (1), 99–103 (in German).—Amidopyrine (**I**) forms with Ni^{2+} and SCN^- a compound of the composition $[Ni(C_{13}H_{17}ON_3)_2](SCN)_2$ which is very stable, slightly soluble in water, ethanol and diethyl ether but soluble in acetone; it is practically insoluble in water in the presence of an excess of the reagent. The reaction is specific for **I** in the presence of phenazone, caffeine, codeine, ethylmorphine, phenacetin, acetanilide, acetylsalicylic acid, barbitone sodium, phenobarbitone sodium, amylobarbitone, ethyl carbamate, atropine, cinchophen, procaine, K guaiacolsulphonate, ephedrine, theobromine, theophylline and lactose. (*i*) *Qualitative test*—Treat the test soln. (if necessary warmed and filtered) with an excess of NH_4SCN (0·5 to 1 g) and then with $Ni(NO_3)_2$ soln. (0·5 to 1 g). Stir vigorously. **I** yields a light-green flocculent ppt. which becomes crystalline. For small amounts of **I** pour the test soln. carefully into a tube containing $Ni(NO_3)_2$ and NH_4SCN soln. A green turbidity will be observed at the interface. After mixing the contents and allowing to stand, a green ppt. is formed. The sensitivity is 30 μg per ml. (*ii*) *Quantitative determination*—Dilute the test soln. (≡0·1 to 0·3 g of **I**, warmed and filtered if necessary) to 20 to 25 ml, add 1 g of NH_4SCN and a soln. of ≃1 g of $Ni(NO_3)_2 \cdot 6H_2O$ in 10 ml of H_2O. Shake well for 3 to 4 min. till a light-green crystalline ppt. is formed. Set aside for 5 to 10 min. and filter through a sintered glass or porcelain crucible. Wash 5 or 6 times with wash-liquor (0·5% of $NiSO_4$ and 0·5% of NH_4SCN), 4 or 5 times with 1 ml of abs. ethanol and 4 or 5 times with 1 ml of diethyl ether. Dry for ≃5 min. by aspiration of air or in a vacuum desiccator and weigh. The determination occupies ≃30 min., and results are accurate to within ±1%.

W. ROUBO

1549. **Application of chelatometry to the determination of nitrogen-containing drugs.** G. Helmstaedter (Wissenschaftl. Abteil. d. Roha-Werks, Bremen-Oberneuland). *Arch. Pharm., Berlin*, 1959, **292** (6); *Mitt. dtsch. pharm. Ges.*, 1959, **29** (6), 91–97.—To determine amidopyrine, 4-*iso*propylphenazone, leptazol, caffeine, theobromine, nicotinamide or the methyl, *iso*propyl or benzyl esters of nicotinic acid, add an excess of $HgCl_2$ soln. to an aq. soln. of the sample (100 or 200 mg), cool the suspension, and collect and wash the ppt.; dissolve it in aq. HCl, add an excess of 0·1 *M* EDTA, render alkaline with aq. NH_3 and titrate with 0·1 *M* $ZnSO_4$. To determine theophylline, use mercuric acetate instead of $HgCl_2$. To determine nicotinic acid, use $CuSO_4$ instead of $HgCl_2$. The average error is $\simeq \pm 1\%$. A. R. Rogers

1550. **Analysis of mandelic acid compounds used in medicine.** G. Helmstaedter (Wissenschaftl. Abteil. d. Roha-Werks, Bremen-Oberneuland). *Dtsch. ApothZtg*, 1959, **99** (25), 589–592.—Mandelic acid may be determined either by cerimetric titration (when it is oxidised to benzaldehyde and CO_2), by acidimetric titration or by conversion into the sparingly soluble copper salt, in which the Cu may be determined by titration with EDTA. The relative errors of the three methods are given for different mandelic acid compounds. M. H. Sawistowska

1551. **Determination of Phenergan [promethazine].** P. Spacu and E. Antonescu (Chem. Res. Centre, Acad. R.P.R., Bucharest). *Acad. R.P.R., Stud. Cercet. Chim.*, 1959, **7** (2), 247–256.—New macro- and micro-gravimetric methods for the determination of promethazine hydrochloride (**I**) are based on the use of $[Cr(SCN)_4(NH_3)_2](NH_4)$ (**II**) and $[Cr(SCN)_6]K_3.4H_2O$ (**III**); **III** is also used for colorimetric and potentiometric methods. *Macro-gravimetric procedure with* **II**—A soln. containing between 0·015 and 0·03 g of **I** is acidified with dil. acetic acid (0·5 to 1 ml) and treated with a 1% soln. of **II** until the supernatant liquid is intensely red. The mixture is heated to 70° to complete the pptn., and is then cooled in ice. The filtered ppt. is washed with water until the filtrate is colourless, and then dried at 110°. *Micro-gravimetric determination*—Between 0·1 and 2 mg of **I** is dissolved, acidified with dil. acetic acid and treated with a slight excess of **II**. It is then mixed well, set aside for 2 to 3 min., and treated as for the macro-gravimetric method. *Procedure with* **III**—A soln. of **I**, acidified with 1 to 2 ml of conc. HCl, is treated with a conc. soln. of **III** until the supernatant liquid becomes violet. (*i*) *Gravimetric method*—The liquid is well mixed, set aside for 5 min., and filtered. The ppt. ($[Cr(SCN)_6]H_3X_3$, where X is promethazine) is then washed with water until free from Cl and dried at 120°. (*ii*) *Potentiometric procedure*—The ppt. is dissolved in acetone ($\simeq$ 20 ml). The ratio of acetone to water should be 1:1 to maintain the complex in soln. The potentiometric titration is carried out at room temp. with a silver electrode and a normal calomel reference electrode, and 0·05 *N* $AgNO_3$. At the end-point 2 to 3 min. must be allowed for equilibrium to be established. (*iii*) *Colorimetric procedure*—The ppt. obtained in (*i*) is dried by suction and dissolved in acetone. The soln. is diluted to 25 or 50 ml with acetone and measured at 574 mμ. H. Sher

1552. **Assay of di-iodohydroxyquin [8-hydroxy-5:7-di-iodoquinoline] by non-aqueous titration.** H. H. Kavarana [British Drug Houses (India) Ltd., Bombay]. *Amer. J. Pharm.*, 1959, **131** (5), 184–187.—*Procedure*—Neutralise the acid impurities in 20 ml of dimethylformamide with 0·1 *N* Na methoxide (1% thymol blue in dimethylformamide or methanol as indicator). Add 8-hydroxy-5:7-di-iodoquinoline (100 to 200 mg) and titrate further with 0·1 *N* Na methoxide to a permanent green colour. The accuracy of the method is improved by titrating in a nitrogen atmosphere or by preventing access of carbon dioxide. Sulphonamides, most barbiturates and certain enols and imides interfere; tablet excipients, kaolin and atropine salts do not. A. G. Cooper

1553. **Spectrophotometric determination of 4-aminosalicylic acid, *m*-aminophenol and *p*-aminophenol.** P. A. Caccia (Veterans Admin. Hosp., Sunmount, N.Y., U.S.A.). *Anal. Chem.*, 1959, **31** (8), 1306–1308.—Sodium nitropentacyanocobaltate[II] reacts with 4-aminosalicylic acid (**I**), *m*-aminophenol and other aromatic compounds having $-NH_2$ and –OH groups *meta* to one another to give an orange compound which obeys Beer's law at 440 mμ in the range of 0 to 75 μg of **I** per ml. The compound obtained with **I** was prepared and its formula and mol. wt. were determined. *p*-Aminophenol is determined in dilute acetic acid soln. with 8-hydroxyquinoline, the extinction being measured at 620 mμ after the addition of NaOH; a suitable concn. range is 1 to 10 μg. The methods can be combined with any general method based on diazotisation and coupling for the analysis of a mixture of *o*-, *m*- and *p*-isomers of primary amines. G. P. Cook

1554. **Method for determining potassium guaiacolsulphonate in pharmaceutical preparations.** P. S. N. Sarma and M. Krishnamurthi (King Inst., Guindy, Madras, India). *Analyst*, 1959, **84**, 522.—The sample is oxidised with HNO_3 and NH_4NO_3 in a Kjeldahl flask. Excess of NH_4NO_3 is removed by further heating, and HNO_3 by evaporation with HCl. The diluted residue is boiled and SO_4^{2-} are pptd. under specified conditions as $BaSO_4$. The collected ppt. is washed with ethanol and diethyl ether, dried by suction and in a vacuum desiccator and then weighed. Results with the salt alone were identical with those obtained by the method of the U.S. National Formulary, and recovery of known amounts from syrups was 100%. A. O. Jones

1555. **Note on the estimation of piperazine and its salts by a turbidimetric method.** R. P. Chakravarti and N. K. Dey (Central Drugs Lab., Gov. of India, Calcutta). *J. Instn Chem., India*, 1959, **31** (2), 53–54.—The soln. of piperazine is diluted to give a concn. of 0·5 g per 100 ml. To this soln. (5 ml) is added Nessler reagent (2 ml) and the vol. is made up to 50 ml. The turbidity is compared in Nessler cylinders against a soln. of pure piperazine. The turbidity can also be measured in a Pulfrich photometer. The results agree with those by the Kjeldahl method. C. A. Slater

1556. **Bromimetric determination of oestrogenic stilbene derivatives.** L. Kraft (Bundesgesundheitsamt, Max-von-Pettenkofer Inst., Berlin-Dahlem). *Arch. Pharm., Berlin*, 1959, **292** (6), 306–314.—Detailed directions are given for the determination of dienoestrol and its diacetate, hexoestrol, and stilboestrol and its dipropionate by reaction with an excess of Br for a specified time, addition of KI and titration with $Na_2S_2O_3$ soln. (*cf.* Gyenes, *Anal.*

Abstr., 1957, **4**, 677). With strict adherence to the directions, the recovery is 100·0 ± 0·3%. The products of bromination of these oestrogenic substances have been characterised.

A. R. ROGERS

1557. **Note on the estimation of zinc oxide in zinc oxide plasters.** A. Bhattacharyya and J. Mukherjee (Central Drugs Lab., Gov. of India, Calcutta). *J. Instn Chem., India*, 1959, **31** (2), 51–52.—Two methods are described. (*i*) The plaster is ignited to const. wt. The residue is dissolved in 0·1 *N* HCl (50 ml), NH_4Cl (1 g) is added and the soln. is titrated with 0·1 *N* NaOH, with methyl orange as indicator. (*ii*) The plaster mass (1 to 2 g) is dissolved in $CHCl_3$ (50 ml). When the plaster has disintegrated, 0·1 *N* HCl (50 ml) is added, followed by NH_4Cl (1 g), and the soln. is titrated with 0·1 *N* NaOH, with methyl orange as indicator. The results agree with those obtained by the B.P.C. 1954 method.

C. A. SLATER

See also Abstracts—**1279**, Analysis of magnesium trisilicate. **1395**, Titration of Cl^- in pharmaceutical compounds. **1409**, Action of bases on $CHCl_3$. **1420**, Inorg. sulphate in Na lauryl sulphate. **1435**, Determination of apiole, piperonal and safrole. **1483**, Griseofulvin in biological fluids. **1484**, Mephenesin in plasma and urine. **1581**, Vitamin A in tablets. **1582**, Vitamin D in multi-vitamin mixtures.

Food

Foods and food additives, beverages, edible oils and fats, vitamins.

1558. **Determination of tin in preserved foods.** A. Porretta and G. Bellucci (Staz. Sper. Ind. Conserve, Parma, Italy). *Ind. Conserve, Parma*, 1959, **34** (2), 107–108.—*Procedure*—The sample (2 to 10 g) is heated in a 100-ml Kjeldahl flask with 10 ml of HNO_3; after carbonisation and cooling, 2 ml of H_2SO_4 is added. The flask is heated and the addition of HNO_3 is continued till the soln. is colourless or slightly yellow. It is then cooled, $HClO_4$ is added and heated to fuming. After cooling, 1 ml of 85% formic acid is added and the soln. is again heated to fuming. *Polarographic determination*—The residue is made up to 25 ml with a soln. *N* in HCl and NH_4Cl; 0·5 ml of 1% gelatin soln is added, and polarography is carried out at −0·3 to −0·8 V and 25°, with a dropping mercury electrode. Lead interferes, but is found in preserved foods in only very low concn, and does not then introduce errors.

L. ZANONI

1559. **Survey of radioactive residues in foods before and after 1945: Evidence of possible fall-out contamination.** E. P. Laug and W. C. Wallace (Food and Drug Admin., Dept. of Health, Education and Welfare, Washington, D.C., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1959, **42** (2), 431–436.—The foods were ashed and the β-activity was measured with an instrument having 100% response to β-particles but a low response to γ-photons; ^{40}K was assumed to be uniformly distributed in nature; its γ-component was ignored. Potassium chloride (100 mg) was used as the working standard. The ash samples were analysed for K (±5%) with a flame photometer, and the contribution of the ^{40}K was deducted from the total activity to give the net activity. Errors are discussed and results tabulated. Fish, shellfish, dairy products and tea (particularly Japanese, Formosan, Malayan and Indian) showed a significant increase in radioactivity when sampled after 1945.

A. A. ELDRIDGE

1560. **Selective enzymatic determination of sugars in sugar factory products.** S. Böttger and W. Steinmetzer. *Z. Zuckerind.*, 1959, **9**, 16–24.—The glucose content is calculated from the amount of O necessary for its conversion into gluconic acid by the use of glucose oxidase; this amount is determined in a Warburg apparatus. This enzymatic determination is specific for glucose and is not influenced by other sugars, *e.g.*, fructose. Oxidation of easily oxidisable constituents can be prevented by adding a small amount of ethanol. Invert sugar can be determined in the same way. Oligosaccharides are determined by first converting them into monosaccharides by means of specific enzymes, *e.g.*, carbohydrases. Procedures are given for the enzymatic analysis of glucose and invert sugar, and also of sucrose and raffinose, by using specific enzymes, in factory products, *e.g.*, molasses and vinasse. The enzymatic analysis has much greater accuracy than polarimetric analysis; optically active non-sugars do not interfere.

SUGAR IND. ABSTR.

1561. **Determination of L-glutamic acid in [beet] sugar factory products.** S. Böttger and W. Steinmetzer. *Z. Zuckerind.*, 1958, **8**, 583–588.—Chemical, chromatographic, microbiological and enzymatic methods of analysis of L-glutamic acid, and their degree of accuracy, are described; the method of enzymatic analysis is considered in detail. A method is given for the analysis of molasses, vinasse, etc., by means of decarboxylase from bacteria (*Clostridium welchii*) or plants (radish). Both carboxylases are specific for the quant. determination of L-glutamic acid, the content of which is calculated from the CO_2 evolved, which is measured in a Warburg apparatus. The determination by radish carboxylase is simpler than by bacterial carboxylase, but is not as quick. Optimum conditions of hydrolysis of L-glutamic acid to pyrrolidone-carboxylic acid have been studied in connection with the manufacture of glutamic acid.

SUGAR IND. ABSTR.

1562. **Quantitative determination of thiols as a constituent of the volatile aromas of bread and baked goods.** H. D. Ocker and A. Rotsch (Bundesforschungsanstalt f. Getreideverarbeitung, Detmold). *Brot u. Gebäck*, 1959, **13**, 165–168.—The thiol content of baked goods (4 to 20 μg per 10 g) depends on the protein content, the baking time and the baking temperature, and is mainly concentrated in the crust. The thiols contribute to the aroma of the baked goods and with good packing survive storage for some days. Thiols were isolated from a thin aq. suspension of the ground sample in a stream of N, collected in a 5% soln. of Hg acetate, and determined by the method of Sliwinski and Doty (*cf. Anal Abstr.*, 1958, **5**, 3515).

E. C. APLING

1563. **Modified Jaffé test for determination of paralytic shellfish poison.** E. F. McFarren, E. J. Schantz, J. E. Campbell and K. H. Lewis (Taft San. Engng Centre, Pub. Health Service, Cincinnati, Ohio, U.S.A.). *J. Ass. Off. Agric. Chem.*, 1959, **42** (2), 399–404.—The Jaffé test as applied to the eluate from a column of XE-64 ion-exchange resin (*cf. Anal. Abstr.*, 1958, **5**, 3896) has been modified

by extracting the unchanged picric acid from the reaction products, after colour development at 38° for 20 min., with ethyl acetate containing 25% of pyridine, and measuring the extinctions at 375 and 490 mμ (the wavelengths of maximum absorption by the interfering material and the shellfish poison, respectively). Standard curves for the absorption of the interfering material and of purified poison are used to derive an equation for calculating the amount of poison present. Satisfactory results were obtained with clams and mussels, but not with Pacific oysters. A. A. ELDRIDGE

1564. **Turbidimetric micro-determination of magnesium in milk.** J. R. Marier and M. Boulet (Nat. Res. Council, Ottawa, Canada). *J. Dairy Sci.*, 1959, **42** (6), 981–988.—The method depends on the formation of a turbidity with K erucate in the presence of K oxalate. *Reagents*—(*i*) Dissolve 2 g of KOH in 6 ml of water and add 60 ml of 95% ethanol. Add 8·5 g of erucic acid, heat gently under reflux for 1 hr. then dilute to 250 ml. (*ii*) When required for use add 1 ml of conc. aq. NH_3 to 6 ml of (*i*) and dilute to 100 ml. (*iii*) Prepare a 30% w/v soln. of K oxalate monohydrate in water. (*iv*) Dilute 2 ml of (*iii*) to 25 ml. *Procedure*—To an aliquot of test soln. (pH 4·0 to 9·0) containing 5 to 40 μg of Mg^{2+} per ml add 8% of its vol. of (*iii*). Set aside for 20 min. then centrifuge at 1000 *g* for 2 min. Place 1·0 ml of the supernatant liquid in a tube and add 4·0 ml of water. In a second tube place an aliquot of a standard Mg^{2+} soln., 1 ml of (*iv*) and water to 5 ml, and in a third tube (blank) 1·0 ml of (*iv*) and water to 5 ml. To all tubes add 5·0 ml of (*ii*) and swirl briskly. Set aside for $\nless$1·5 hr. and read the extinctions at 400 mμ. The results agree within $\pm$3·3% with those given by a standard ashing procedure. W. H. C. SHAW

1565. **Direct spectrophotometric determination of butylated hydroxyanisole in lard and in hardened lard.** P. V. Hansen, F. L. Kauffman and L. H. Wiedermann (Swift Co., Chicago, Ill., U.S.A.). *J. Amer. Oil Chem. Soc.*, 1959, **36** (5), 193–195.—The fat ($\simeq$25 g) is melted on a steam bath, and to it are added 10 ml of $CHCl_3$ and 100 ml of methanol; after being heated almost to boiling and then shaking for 1 min. the mixture is chilled in an ice bath and filtered (Whatman No. 1), the residual fat being further extracted twice with 5 ml of $CHCl_3$ and 50 ml of methanol. The combined extracts are concentrated to 10 ml, chilled and filtered, and the filtrate is finally diluted to 25 ml with methanol. Spectrophotometric determinations are then made at 290 mμ, with a 1-cm silica cell, and the butylated hydroxyanisole content is obtained by reference to a standard curve. Butylated hydroxytoluene and tocopherols interfere. The method is more reliable than existing colorimetric procedures, especially for low concn. of antioxidant. G. R. WHALLEY

1566. **Gravimetric estimation of vanillin via its semicarbazone.** K. K. Kaistha (State Drug Control Lab., Ambala Cantt., Punjab, India). *Drug Standards,* 1959, **27** (5), 141–142.—The method described previously for the determination of camphor (*cf. Anal. Abstr.*, 1957, **4**, 4108; 1959, **6**, 1905) is now used for vanillin; the reagents are heated under reflux for 40 min. at 70°. The method is preferred to the volumetric method of the B.P. 1948, Appendix I, which gives an indefinite end-point, and to gravimetric dinitrophenylhydrazine methods, which give high results. A. R. ROGERS

1567. **Isolation and quantitative determination of oil-soluble colours in foods.** R. S. Silk (Div. Cosmetics, Food and Drug Admin., Dept. of Health, Education and Welfare, Washington, D.C., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1959, **42** (2), 427–430.—Adsorption chromatography, first with Al_2O_3 and then with a mixture of MgO and Celite 545, is employed to isolate the following dyes from fats and oils and to eliminate carotenes and annatto—FD & C Yellow 3 (C.I. Food Yellow 10), FD & C Yellow 4 (C.I. Solvent Yellow 5); D & C Red 17 (C.I. Solvent Red 23), D & C Red 18 (C.I. Solvent Red 27), D & C Green 6 (C.I. Solvent Green 3), D & C Violet 2 (C.I. Solvent Violet 13), D & C Yellow 11 (C.I. Solvent Yellow 33); Ext. D & C Orange 4 (C.I. Solvent Orange 2), Ext. D & C Red 14 (C.I. Solvent Orange 7); aminoazobenzene (C.I. Solvent Yellow 1); aminoazotoluene (C.I. Solvent Yellow 3); dimethylaminoazobenzene (C.I. Solvent Yellow 2); phenylazo-2:4-resorcinol (C.I. Food Orange 3); and phenylazo-2-naphthol (C.I. Solvent Orange 14). Colours are then tentatively identified by paper partition chromatography, and then identified by column partition chromatography by using silicone-treated Celite that has been treated with soya-bean oil (5%) in diethyl ether. With the column procedure described, the order in which the colours are eluted is the same as that obtained by paper chromatography. A. A. ELDRIDGE

1568. **Artificial colouring materials excluded from use in foodstuffs. I.** C. Calzolari and E. Cerma (Trieste Univ.). *Chim. e Ind.*, 1959, **41** (5), 425–430.—Methods are being examined for the determination of 17 colouring materials, recently banned in Italy for use in foodstuffs. The initial report deals with the polarographic determination in the pure form of Naphthol yellow (C.I. Food Yellow 1), Aniline yellow (C.I. Solvent Yellow 1), Chrysoidine, Orange 1 (C.I. Acid Orange 20), Acid Bordeaux, Ponceau 2R (C.I. Food Red 5), Biebrich scarlet, eosin (C.I. Acid Red 87) and phloxin (C.I. Acid Red 98). L. A. O'NEILL

1569. **Detection and identification of antimicrobials by paper chromatography.** C. Genest and R. A. Chapman (Food and Drug Div., Dept. of Nat. Health and Welfare, Ottawa, Canada). *J. Ass. Off. Agric. Chem.*, 1959, **42** (2), 436–440.—Antimicrobial substances likely to be found in foods may be separated and identified by paper chromatography. Three systems are described. In system (*i*) the stationary solvent is formamide in acetone, the mobile solvent is acetic acid in $CHCl_3$, and the chromogenic agents are $KMnO_4$ in aq. Na_2CO_3, $FeCl_3$, and $AgNO_3$ in aq. ammoniacal ethanol; *p*-hydroxybenzoic acid, salicylic acid, benzoic acid, sorbic acid and dehydracetic acid are detected. In system (*ii*) the stationary solvent is H_2O, the mobile solvent is ammoniacal aq. acetone, and the chromogenic agents are Universal Indicator, phthalic acid and aniline in H_2O and *n*-butanol, and $PtCl_4$ in KI soln.; sodium diacetate, propionic acid, hexamine and Na_2SO_3 are detected. In system (*iii*) the stationary solvent is formamide in acetone, the mobile solvent is triethylamine in cumene, and Millon's reagent or illumination by ultra-violet light is employed for locating the spots; methyl, ethyl, propyl, butyl and benzyl *p*-hydroxybenzoates are thus detected. A. A. ELDRIDGE

1570. **Determination of cacao shell in cacao products from the galacturonic acid content, and the effect of alkalisation.** C. J. Reeskamp (Cacaofabriek "De Zaan," Koog-Zaandijk, Holland).

J. Ass. Off. Agric. Chem., 1959, **42** (2), 408–410.—The determination of cacao shell by means of Winkler's galacturonic acid method (*Ibid.*, 1957, **40**, 328) is of doubtful value on account of the great differences in pectin content exhibited by cacao shell. Alkalisation renders the pectin less soluble, thus affecting the results obtained by gravimetric pectic acid determinations. A. A. ELDRIDGE

1571. **Determination of extract in experimental malting barleys by new micro-mashing and falling-drop methods.** O. J. Banasik and R. H. Harris. *Wallerstein Labs Commun.*, 1959, **22**, 81–88.—A micro-mashing procedure is described in which 3·5 g of ground barley is mashed in a specified manner with 25 ml of a soln. containing 0·19% of α-amylase and 0·45% of malt diastase. The filtrate is brought to 20°, and the time taken for a drop to fall between two reference lines on a glass tube, maintained at 20° and containing a mixture of bromobenzene and kerosine (approx. 2:5), is measured, and compared with the times taken by drops of soln. of $CuSO_4$ of known sp. gr. The procedures for introducing the drops, adjusting the sp. gr. of the bromobenzene - kerosine mixture, timing the fall, and constructing the standard curve are similar to those of Clendinnen (*Ibid.*, 1958, **21**, 24). The results agree well with those obtained by pyknometer. J. INST. BREW. ABSTR.

1572. **Tannin determination in beer and wort by ultra-violet spectrophotometry.** W. Kleber and P. Schmid (Wissenschaftl. Sta. f. Brauerei, München e.V., Germany). *Brauwissenschaft*, 1959, **12** (5), 117–119.—The method of Owades *et al.* ("Determination of Tannins in Beer and Brewing Materials by Ultraviolet Spectrophotometry," Schwarz Labs., Inc., N.Y.) was used. Some of the processes used for separating the tannins were modified and tannin values found for beer and wort are reported. The u.v. spectrophotometric method was more satisfactory than the method of pptn. with cinchonine sulphate. S.C.I. ABSTR.

1573. **Chemical determination of alcohol in wines and stillage by dichromate.** J. F. Guymon and E. A. Crowell (Dept. of Viticulture and Enology, Univ. California, Davis, U.S.A.). *J. Ass. Off. Agric. Chem.*, 1959, **42** (2), 393–398.—The method of Flanzy *et al.* (*Ann. Tech. Agr., Paris, E*, 1955, **4**, 397) was slightly modified by employing a standard $K_2Cr_2O_7$ solution containing 33·768 g per litre to correspond with the U.S.A. definition of % alcohol by vol. at 60° F. The method is precise and accurate. With pyknometric results as a standard, the mean and standard deviations (in % by vol.) were −0·059 and ±0·125, respectively, by the wine hydrometer method, and +0·008 and ±0·069, respectively, by the chemical method, for 16 wines.
A. A. ELDRIDGE

1574. **Iodine value of the unsaponifiable matter in the differentiation of olive from other oils.** L. Frontero (R.I.O., Imperia, Italy). *Olii Min.*, 1959, **36** (5), 154–156.—The iodine value of the unsaponifiable matter of olive and other oils differed considerably according to the procedure used for isolating the unsaponifiable matter. By using the same method of isolation, expressed olive oils and "rectified A" oils could be differentiated by this property from "rectified B" oils, esterified oils, other vegetable oils and esterified animal fatty acids.
L. A. O'NEILL

1575. **Determination of fatty acid composition by gas chromatography and u.v. spectrophotometric methods.** L. Malin. *Lab. Practice*, 1959, **8** (6), 226–228.—Gas chromatography of the mixture of methyl esters, and u.v. spectrophotometry of the mixture of unsaturated fatty acids after isomerisation to the conjugated acids, are useful methods of analysis. The isomerisation is achieved by heating the sample with KOH and glycerol at 180°. In ethanol soln. both diene and triene conjugated acids have an absorption max. at 232 mμ, while only the trienes absorb at 268 mμ. G. BURGER

1576. **Glass-[fibre] paper chromatography of the long-chain fatty acids, brominated derivatives and methyl esters.** R. L. Ory, W. G. Bickford and J. W. Dieckert (S. Regional Res. Lab., U.S. Dept. of Agric., New Orleans, La., U.S.A.). *Anal. Chem.*, 1959, **31** (8), 1447–1448.—Saturated and unsaturated fatty acids may be easily separated by bromination of their methyl esters and subsequent chromatography on glass-fibre paper impregnated with silicic acid, with *iso*octane as the developing solvent.
K. A. PROCTOR

1577. **Ricinelaidic acid and methyl ricinelaidate; their preparation and determination by infra-red spectroscopy.** M. A. McCutchon, R. T. O'Connor, E. F. Dupre, L. A. Goldblatt and W. G. Bickford (Southern Reg. Res. Lab., New Orleans, La.). *J. Amer. Oil Chem. Soc.*, 1959, **36** (3), 115–118—. The i.r. spectra of these two compounds are presented and bands arising from a deformation of the C–H about the *trans* C=C group are discussed. Absorptivity values for this band in $CHCl_3$ and CS_2 soln. are given as additional criteria of purity. The i.r. absorption procedure for the quant. determination of the isolated *trans* ethylenic bond was applied to the determination of both ricinelaidic acid and its methyl ester. Details of methods for the determination of each compound are given with the necessary equations. The error of the determination of methyl ricinelaidate in mixtures with methyl ricinoleate is $\not>$1% (relative). N. E.

1578. **Isolation, examination and determination of polyoxyethylene glycol (Carbowax) surface-active agents added to edible fats.** S. Anselmi, L. Boniforti and R. Monacelli (Ist. Sup. di Sanità, Rome). *Chim. e Ind.*, 1959, **41** (5), 421–424.—The fat is saponified and acidified, the fatty acids are removed by extraction and the aq. phase containing the polyoxyethylene glycol(**I**), glycerol, etc., is examined. **I** is identified by the addition of a soln. of iodine in KI which gives a ppt. the colour of which depends on the mol. wt. The content of **I** may be determined by separating this complex, dissolving it in KI and acetone and determining its iodine content by titration with $Na_2S_2O_3$ soln. The mol. wt. of the **I** present may be determined by infra-red spectroscopy, by extracting it from the evaporated aq. soln. with benzene and examining the intensity of the –OH absorption in the 2 to 3-μ region.
L. A. O'NEILL

1579. **Paper-chromatographic separation of some antioxidants.** B. A. Dehority (Agric. Expt. Sta., Storrs, Conn., U.S.A.). *J. Chromatography*, 1959, **2** (4), 384–387 (in English).—Three techniques are described for the separation of 8 food antioxidant additives. Of these, propyl gallate, nordihydroguaiaretic acid (NDGA), butylated hydroxyanisole (BHA) and butylated hydroxytoluene (BHT) are

completely separated on paper treated with cotton-seed oil, with acetone - ethyl acetate - water (3:1:6) as solvent. The values of R_{PG} (distances moved relative to propyl gallate) are given for the 8 compounds in each of the three systems.

G. BURGER

1580. **Analytical detection of synthetic antioxidants in edible fats. II. Separation and identification of synthetic antioxidants by means of thin-film chromatography.** A. Seher (Dtsch. Inst. f. Fett-Forsch., Münster, Westf.). *Fette, Seif., Anstrichmitt.*, 1959, **61** (5), 345–351.—Synthetic antioxidants can be satisfactorily chromatographed by the thin-film method, in which a layer of silica gel containing a little gypsum, and fixed to a glass plate or strip, is used as adsorbent. The spots can be detected by means of molybdophosphoric acid (20%) in ethanol or 2-methoxyethanol. All the 24 compounds examined gave positive reactions except α-tocopheryl acetate; the monoglyceride of citric acid needed warming to 120° to develop the colour. 2:6-Dichloro-*p*-benzoquinone-4-chlorimine is a less satisfactory reagent. Both of these reagents react only with unchanged antioxidants. For the chromatography no solvent is entirely satisfactory, but two-dimensional chromatography, with $CHCl_3$ followed by benzene, gives a good separation. It is best to apply three spots, and after development two are eluted with one solvent each and the third is chromatographed in two dimensions. The evaluation of the chromatograms by means of templates, the experimental factors influencing the R_F values, the photographic recording of the chromatograms and results obtained by examining commercial antioxidants are discussed in detail.

A. G. PETO

1581. **A partition column for vitamin-A chromatography.** J. B. Wilkie, S. W. Jones and W. W. Morris, jun. (Div. Nutrition, Food and Drug Admin., Dept. of Health, Education and Welfare, Washington, D.C., U.S.A.). *J. Ass. Off. Agric. Chem.*, 1959, **42** (2), 422–423.—The partition system in which a polyoxyethylene glycol 600 is used as the stationary phase with Celite 545 as support and *iso*octane as the mobile phase (Theivagt and Campbell, *Anal. Chem.*, 1959, **31**, 1375) is improved by the use of light petroleum (boiling-range 30° to 60°) as a supplement in the mobile phase. Details of procedure are given. Acceptable results were obtained for recovery and reproducibility in the determination of vitamin A in tablets.

A. A. ELDRIDGE

1582. **Determination of vitamin D in multivitamin mixtures after separation by partition chromatography.** J. G. Theivagt and D. J. Campbell (Anal. Res. Dept., Abbott Lab., N. Chicago, Ill., U.S.A.). *Anal. Chem.*, 1959, **31** (8), 1375–1377.—Mixtures with vitamin A to vitamin D ratios as high as 50 to 1 have been separated and the results on a wide variety of samples agree closely with those of the official U.S.P. biological assay. Celite mixed with a polyoxyethylene glycol and *iso*octane is used for the column packing, and *iso*octane as the mobile phase, and duplicate assays can be completed in 6 hr. In some low-potency samples an extraction of the vitamin D before saponification is required. In triplicate analyses on multivitamin tablets with minerals the coeff. of variation was ±1·6%.

K. A. PROCTOR

1583. **Combined method for vitamin-C assay in biological and food products.** K. A. Bogdański (Politech. Coll., Łódź, Poland). *Roczn. Technol. i Chem. Żywności*, 1958, **3**, 21–50.—A method for the bacterial-enzyme photometric determination of the vitamin-C content of biological and food products is developed, which combines the Stewart and Sharp enzyme-microbiological method (*Ind. Eng. Chem., Anal. Ed.*, 1945, **17**, 373), as modified by Bogdańska *et al.* (*Roczn. Państw. Zakl. Hig.*, 1954, **5**, 327), and the U.S. Vitamin Chem. Assn. photometric technique ("Methods of Vitamin Assay," Interscience Publ., N.Y., 1951). The new method permits quant. determinations of ascorbic acid (**I**), dehydroascorbic acid (**II**), and the two types of compound usually responsible for high results in the usual oxidation methods. For max. accuracy the bacterial count in the sample should not exceed 3 million living cells per μg of **I**. The accuracy largely depends on the recovery of **I** from **II**, and a suitable strain of *Escherichia coli* of a high reducing capacity ((ATCC No. 9492) is required. In certain colorimeters the units in which the light-transmission scale is graduated are rather large; in such cases the accuracy can be increased by repeating the photometric determinations 3 to 5 times.

A. GROCHOWSKI

See also Abstract—**1479**, Identification of α-activity of wheat.

Sanitation

Analysis of air, water, sewage, industrial wastes, industrial poisons.

1584. **Photometric determination of esters of acetic acid in the atmosphere.** V. Šedivec (Inst. Hyg., Ind. Diseases, Prague). *Pracovní Lékařství*, 1959, **11** (5), 245–249.—Esters of acetic acid are treated with hydroxylamine and the hydroxamic acid so formed is determined photometrically after reaction with Fe^{3+}. *Procedure*—Draw the air (5 litres) through a train of three flasks containing *iso*propyl alcohol (10 ml) (cooled with ice for methyl or ethyl acetate). Transfer the content of each flask to a 25-ml flask, and rinse the flasks with *iso*propyl alcohol (5 ml); combine the liquids and add hydroxyammonium chloride soln. (10%) (1 ml) and 2·5 *N* NaOH (2 ml) and set aside for 20 min. Add $FeCl_3$ soln. (27 g of $FeCl_3.6H_2O$ dissolved in 300 ml of 2·5 *N* HCl and diluted to 500 ml), mix, dilute to vol. with H_2O and measure the extinction of an aliquot at 530 mμ. Compare with a calibration curve. The method is suitable for the determination of 0·2 to 20 mg of ester.

J. ZÝKA

1585. **Detection and estimation of air-borne proteins by pyrolysis to hydrogen cyanide.** C. O. Thomas and B. B. Baker (S. Res. Inst., Birmingham, Ala., U.S.A.). *Anal. Chem.*, 1959, **31** (8), 1391–1393.—Apparatus is described for the collection of protein material from air, subjecting it to pyrolysis at 500°, and detection of the cyanide formed by the electrochemical method of Baker and Morrison (*Ibid.*, 1955, **27**, 1306).

H. F. W. KIRKPATRICK

1586. **Modified 4:7-diphenyl-1:10-phenanthroline method sensitive to 1 p.p.b. [U.S.A.] of iron in high-purity water.** W. G. Knapp (Argonne Nat. Lab., Lemont, Ill., U.S.A.). *Anal. Chem.*, 1959, **31** (8), 1445–1446.—This modification of an earlier method

(*cf.* Smith *et al.*, *Analyst*, 1952, **77**, 418) permits the rapid determination of 1 part of Fe in 10^9 parts of high-purity H_2O. The time required when six or more samples are run simultaneously is <5 min. per determination. *Procedure*—Mix the sample (100 ml) with 10% hydroxyammonium chloride soln. (2 ml) and 4:7-diphenyl-1:10-phenanthroline soln. (0·001 *M* in 50% ethanol) (10 ml). Extract with purified *n*-hexanol (5 ml) for 10 min, discard the aq. layer, and compare the organic phase with standards by axial viewing. For higher Fe contents dilute the alcohol extract or use a spectrophotometer set at 533 mμ. J. P. STERN

1587. **Determination of trace elements in water by neutron activation analysis.** R. L. Blanchard and G. W. Leddicotte (Oak Ridge National Lab., Tenn.). *U.S. Atomic Energy Comm.*, Rep. ORNL-2620, 1959, 78 pp.—Neutron activation analysis was applied to seven water samples from selected geographical areas in the U.S. The technique proved adequate for the identification and measurement of 19 elements in concn. from 0·00002 to 63·3 p.p.m. The samples were irradiated and elements were identified by γ-spectrometry and the measurement of specific induced radionuclides. The degree of chemical separation required for a given sample depended on the element being determined and the variety of constituents present. It was possible to determine Cl, Na and A on the sample as irradiated. Most other elements required separation. Ion exchange and pptn. were investigated, and pptn. proved most generally satisfactory. General chemical separation was adequate for certain elements; others required specific procedures. A summary of the elements identified, time of irradiation and analytical procedure is given. The results suggest that nuclear activation analysis is of value in sanitary engineering and geochemical water studies. The technique is specific in that each induced radionuclide decays with its own characteristic radiation and half-life. It is sensitive in that amounts as small as 10^{-11} g of some elements can be determined with an accuracy of ±5% or better. The study was limited to elements yielding radionuclides with half-lives ranging from minutes to days. The location of counting equipment nearer to the irradiation facilities would permit determinations involving radionuclides of much shorter half-lives to be made, thus widening the range of elements suitable for analysis. NUCL. SCI. ABSTR.

1588. **Identification of small quantities of fluorine in waters rich in chloride.** F. Moreno and M. Vila (Dept. Invest. Bromatológ., Barcelona). *An. Bromatologia*, 1959, **11** (2), 185-187.—The method consists in treating the dry residue containing F^- and Cl^- with water (10 ml) and H_2SO_4 (15 ml), stirring to liberate HCl gas, and adding Ag_2SO_4 until the pptn. of AgCl is complete. The filtrate is then distilled until the temp. rises to 160°; the distillate is neutralised with NaOH and evaporated to dryness in a 10-ml porcelain crucible. Fluoride is detected by treatment of the residue with 90% H_2SO_4 and a little powdered glass, and collection of the volatilised SiF_4 in a drop of water suspended on a flat watch-glass. Evaporation of the drop and cleaning of the glass leaves an etched mark in the presence of F^-; 0·01 to 0·1 mg can be identified with certainty in the presence of 1 g of NaCl. E. C. APLING

1589. **Polarographic determination of mercury in brines and industrial waters.** F. Grandi and L. Salvagnini (Sicedison S.p.A., Venice, Italy). *Chim. e Ind.*, 1959, **41** (5), 430-431.—Mercury ions in the systems studied show a polarographic reduction wave at about zero potential. The effective increase in current produced by the diffusion of mercury ions to the dropping mercury electrode is approx. proportional to their concn. Brines, which may be chlorinated to various degrees, are first saturated with Cl and then reduced with a known amount of $NaAsO_2$. For industrial waters circulating in plants for the electrolytic production of NaOH, this step may be omitted if Cl is absent. The mercury ions are then determined polarographically, after saturation of the materials with NaCl. L. A. O'NEILL

1590. **Removal of cyanides from industrial wastes by chlorination. II. Analytical methods.** B. Balla and T. Bene (Res. Inst. for Heavy Chem. Ind., Veszprém, Hungary). *Nehézvegyipari Kutató Intézet Közleményei*, 1959, **1** (3-4), 199-206.—Methods were developed for the determination of CN^-, OCN^-, CNCl and active chlorine in the decyanisation process. Cyanide contents >0·5 mg per litre can be titrated with 0·01 *N* iodine by using the dead-stop method at 0·15 V. When other reducing components are also present, the HCN is first steam-distilled from the acidified soln. into alkali. Traces of CN^- can be determined by the pyrazolone-pyridine method of Dodge and Zabban (*Plating*, 1953, **42**, 71); a modification of this process is suitable for the determination of CNCl. Cyanates are hydrolysed at pH 2 in 15 min. and the NH_3 produced is determined photometrically with Nessler reagent. The active chlorine content is measured by adding an excess of arsenite at pH 11 to 12 and titrating the excess with 0·1 *N* iodine. When only small quantities are present, 0·01 *N* iodine and the dead-stop method should be used. G. SZABO

See also Abstracts—**1263**, Potassium in sea water. **1264**, Potassium, Rb and Cs in sea muds. **1348**, Rhenium in industrial wastes. **1397**, Organic matter in water residues. **1597**, Calibration of pumps for air sampling.

Agricultural analysis

Soil, fertilisers, herbicides, pesticides, animal feeding-stuffs.

1591. **Chromatographic separation of pentose and hexose in hydrolysates.** Yu. I. Brid'ko. *Sb. Stud. Nauch. Rabot. Kubansk. S.Kh. Inst.*, 1957 (1958), (2), 149-152; *Ref. Zhur., Khim.*, 1959, (18), Abstr. No. 64,253.—The chromatographic separation previously described [*Ref. Zhur., Khim., Biol. Khim.*, 1957, (22), 24,101] is modified for the separation of pentoses and hexoses in the hydrolysates obtained from agricultural waste products by using the radial method, which can separate 8 sugars simultaneously. The separation is effected on paper circles (12 cm in diam.) in Petri dishes. Drops (0·005 μl) of the soln. (1 to 2%) are placed on the starting line; the chromatogram is developed with the upper layer of a mixture of *n*-butanol, glacial acetic acid and water (13:1:6) (I) or ethyl acetate, water and pyridine (5:5:1) (II). After 80 min. (for I) or 30 min. (for II), the chromatogram is dried, cut into sectors, and treated with the reagent (10 ml of

96% ethanol, 0·4 g of salicylic acid and 0·5 mg of *o*-toluidine), which after heating at 100° to 105° for 3 to 5 min. gives a cherry-red colour with pentoses and a dirty green colour with aldohexoses. The presence of furfuraldehyde in the soln. hinders the development of the spots, so that it is then recommended to dry the chromatogram for 24 hr. in air at $\simeq$20° before application of the reagent. C. D. KOPKIN

1592. **Determination of phosphates in soil by the phosphorus-32 isotopic dilution method.** C. Sapetti (Staz. Chim. Agr. Sper., Torino, Italy). *Ann. Sper. Agr.*, 1959, **13** (1), 99–106.—*Procedure*—The soil (1 to 5 g, containing $\simeq$4 mg of P_2O_5) is treated with 20 ml of conc. HCl and 5 ml of 5 *N* HNO_3 after the addition of 1 ml of ^{32}P soln. ($\simeq$0·5 μC per ml); 10 ml of H_2SO_4 - $HClO_4$ is added (200 ml of $HClO_4$ and 220 ml of H_2SO_4 made up to 1 litre with H_2O) and the soln. is evaporated to white fumes. The cooled residue is treated with 50 ml of 5 *N* HNO_3, heated and filtered, collecting 30 ml. This filtrate is heated and treated with ammonium molybdate reagent [dissolve 25 g of $(NH_4)_2SO_4$ in 250 ml of HNO_3 (sp. gr. 1·36), and 75 g of ammonium molybdate in 250 ml of H_2O, mix, and filter after 2 days]. The soln. is shaken and set aside for 10 min. at 60° to 70°. The ppt. is separated by centrifuging and washed with 2% HNO_3, H_2O and twice with acetone, and is dried *in vacuo*; $\simeq$50 mg is weighed into a counting plate, homogenised with 2 drops of acetone, and counted for 2 min. The standard is prepared by adding 0·5 ml of the ^{32}P soln. to 50 mg of inactive ammonium molybdophosphate in a counting plate and evaporating to dryness. This is stable for a month. L. ZANONI

1593. **Colorimetric estimation of malathion residues in cottonseed.** M. V. Norris and E. J. Kuchar (American Cyanamid Co., Stamford, Conn.). *J. Agric. Food Chem.*, 1959, **7** (7), 488–489.—The colorimetric method previously described (Norris *et al.*, *Ibid.*, 1958, **6**, 111) is adapted for use on cottonseed by Soxhlet extraction of the oil and residues with hexane, the extraction of the residues into methyl cyanide, passage through a column of acid-washed alumina to remove coloured material, and extraction into CCl_4, followed by the procedure previously described. M. D. ANDERSON

1594. **Micro-determination of TDE [1:1-dichloro-2:2-di-(*p*-chlorophenyl)ethane] in spray residues.** I. Rosenthal, C. F. Gordon and E. L. Stanley (Rohm and Haas Co., Philadelphia, Pa., U.S.A.). *J. Agric. Food Chem.*, 1959, **7** (7), 486–488.—The insecticide TDE is determined in plant extracts (after partial purification by solvent partitioning into methyl cyanide, treatment with adsorbent, and removal of HCl) by reaction with Na ethoxide in dimethylformamide to yield 1-chloro-2:2-di-(*p*-chlorophenyl)ethylene, which on treatment with H_2SO_4 gives a coloured carbonium ion complex with max. absorption at 502 mμ. The method permits the detection of 1·0 μg; the recovery of TDE is 62 to 65%. M. D. ANDERSON

1595. **Quantitative determination of the principal biologically active constituent in Delnav, an organophosphorus insecticide.** P. Casapieri and H. H. Keppler (Res. Dept., African Explosives and Chem. Industries Ltd., P.O. Northrand, Transvaal, S. Africa). *J. S. Afr. Chem. Inst.*, 1959, **12**, 26–28.—A method is described for the determination of 2:3-*p*-dioxandithiol SS-bis-(OO-diethyl phosphorodithioate) (**I**), which is the main constituent in Delnav and in spray-washes containing it. *Procedure*—Apply uniformly 0·2 μl of a 0·5% soln. of Delnav in hexane along a base line on each of 5 sheets of Whatman No. 2 paper (27·5 cm square) previously impregnated with liquid paraffin from a 5% soln. in hexane, and develop the chromatograms for 12 hr. by the ascending technique with a solvent consisting of the upper phase of a mixture of $CHCl_3$, ethanol and water (5:5:3). After drying, spray one chromatogram with 0·2 *M* $HgCl_2$, followed immediately by 0·08 *M* 2:4-dinitrophenylhydrazine in 32% $HClO_4$, to locate the position of **I** as a brown area ($R_F \simeq$ 0·05), and then cut strips containing the **I** from the remaining unsprayed sheets. Extract the **I** from each strip separately with methanol (20 ml) for 2 hr. in a Soxhlet apparatus, evaporate off the solvent and determine the P content of the residues of **I** and liquid paraffin by the molybdovanadate method (*cf.* Koenig and Johnson, *Ind. Eng. Chem.*, *Anal. Ed.*, 1942, **14**, 155), with $HClO_4$ in a concn. of 0·2 to 1·4 *M* in the final soln. and allowing 1 hr. for full colour development. A synthetic sample of **I** gave a recovery of 94% ($\pm$3% on 14 determinations) by the chromatographic procedure; from the P content of an unchromatographed sample, a figure of 103% ($\pm$1·5% on 9 determinations) was obtained, indicating that thio esters of higher P content were probably present. For the analysis of spray-washes, break the emulsion by adding 1 g of $Al_2(SO_4)_3$ to 50 ml of the preparation (nominally containing 0·1% of Delnav), extract the mixture with three 25-ml portions of hexane, evaporate the combined extracts to 2 to 3 ml, and dilute to 10 ml with hexane before chromatographing an aliquot as described above. The **I** content of Delnav was found to be 75% $\pm$ 4% and this factor was used to convert **I** to Delnav in the analysis of spray-washes. S. C. JOLLY

See also Abstracts—**1494**, **1495**, **Determination of gibberellins.**

5.—GENERAL TECHNIQUE AND APPARATUS

General

1596. **Constant-head aspirator.** S. R. Howarth (Safety in Mines Res. Estab., Sheffield, England). *J. Sci. Instrum.*, 1959, **36** (6), 291.—A simple, portable and robust apparatus is described for drawing air through an instrument at constant flow rates between 1 and 10 cu.cm per min. The average flow-rate in successive 15-min. periods remains constant within $\pm$4% over a period of 9 hr. P. T. BEALE

1597. **Calibration of hand pumps for air sampling.** J. C. Gage (I.C.I. Ltd., Industrial Hygiene Res. Lab., Welwyn, Herts., England). *Analyst*, 1959, **84**, 519–520.—The simple apparatus described is made with ordinary laboratory apparatus, *viz*, a water aspirator and glass and rubber tubing. It gives a direct reading, in the form of a vol. of water, of the vol. of the air sample taken at atm. pressure under the conditions of use. A. O. JONES

1598. **A constant delivery-rate burette.** J. R. Gwilt and A. Robertson (Winthrop Laboratories Ltd., Newcastle upon Tyne, England). *Chem. & Ind.*, 1959, (35), 1088.—A standard 100-ml burette with a tap at the top and an on/off and a fine-control tap at the bottom is illustrated. The capillary side-arm which is introduced below the normal lowest level of titrant keeps the liquid at this level at atmospheric pressure during operation: the top tap is closed, the on/off tap is opened and the fine-control tap is adjusted to a satisfactory rate of flow. The hydrostatic head, that is, the distance between the tip of the capillary air leak and the tip of the burette delivery jet, is then constant.
E. G. CUMMINS

1599. **Method of, and apparatus for, the titration of solutions and the like purposes.** J. E. Dubois. Brit. Pat. 824,266; date appl. 29.12.55. France, date appl. 31.12.54.—Two (platinum) electrodes are immersed in the solution to be titrated and a direct potential higher than the highest potential of any transformation occurring during the titration (2·8 to 3·2 V) is applied between the electrodes through a resistor. The changes that occur in the potential between the two electrodes during the titration are amplified and used to indicate or control the course of the titration.
J. M. JACOBS

1600. **Delay circuit for fraction collection of very volatile liquids.** W. H. Bancroft, jun. (Veterans Admin. Hosp., Birmingham, Ala., U.S.A.). *Anal. Chem.*, 1959, **31** (8), 1447.—The device described overcomes the problems of evaporation when a drop-count collector is used, and of differing rates of collection when a preset-time collector is used It consists of an intermittent calibrated siphon used in conjunction with a delay circuit in series with the drop counter.
K. A. PROCTOR

1601. **Viscosity of fluids at high pressures. Rotating cylinder viscometer and the viscosity of *n*-pentane.** H. H. Reamer, G. Cokelet and B. H. Sage (Chem. Engng Lab., California Inst. of Technol., Pasadena, U.S.A.). *Anal. Chem.*, 1959, **31** (8), 1422–1428.—A rotating cylinder viscometer suitable for measurements at pressures up to 25,000 p.s.i. in the temp range 0° to 500° F is described. The results obtained with *n*-pentane are in good agreement with these obtained by other workers.
K. A. PROCTOR

Chromatography, ion exchange, electrophoresis

1602. **Feeding device for step-wise gradient elution in column chromatography.** O. Mikeš, V. Tomášek and V. Holeyšovský (Biochem. Dept., Chem. Inst., Acad. Sci., Prague). *Chem. Listy*, 1959, **53** (6), 609–613.—A pipetting apparatus is described which permits the systematic continuous addition of a series of buffered soln. to the column. By the action of compensation effects in the column the steps are eluted in a continuously linear gradient. The addition and measurement of the added buffer soln. are carried out automatically.
J. ZÝKA

1603. **Apparatus suitable for applying fairly large quantities of solutions on paper chromatograms.** D. van der Sijde and J. de Flines (Royal Netherlands Fermentation Industries, Ltd., Delft). *J. Chromatography*, 1959, **2** (4), 436–437 (in English).—Van Gulik's apparatus (*Nature*, 1956, **178**, 994) has been modified by the addition of a drying system by which a current of warm air is passed round the spot from below the paper. With an air flow of 60 litres per min. at 110°, 0·5 ml of a steroid soln. in 4-methylpentan-2-one can be applied as a spot of 1 cm diam. in 2 min. Detailed illustrations of the apparatus are given.
G. BURGER

1604. **Improved resolution on paper chromatograms.** L. I. Woolf (Hosp. for Sick Children, London). *J. Chromatography*, 1959, **2** (4), 431–432 (in English).—A sample soln. spotted on to chromatographic paper spreads to a small disc. If the spot is dried and small portions of the solvent are applied to the centre, the solute forms a ring. For R_F values <0·4 this ring formation is retained on running the chromatogram. Overlapping rings are more easily resolved than overlapping solid spots. The method fails for substances with high R_F values.
G. BURGER

1605. **Ionic phenomena which modify separation in paper chromatography.** F. Miranda and S. Lissitzky (Fac. de Méd. et Pharm., Marseille, France). *J. Chromatography*, 1959, **2** (4), 354–361 (in French).—Anomalies observed in a chromatographic study of mono-substituted guanidine derivatives in natural extracts (*e.g.*, of scorpion muscle) are attributed to ionic changes caused by the paper, the solvent and the electrolytes present. The sensitivity of the material to these influences is due to its non-amphoteric character.
G. BURGER

1606. **Monochromatic ultra-violet light source for the evaluation of chromatograms.** K. Štefka and J. Soška (Biochem. Inst., Acad. Sci., Brno, Czechoslovakia). *Chem. Listy*, 1959, **53** (6), 613–614.—Monochromatic u.v. radiation of wavelength 254 mμ is obtained when the light from a mercury vapour lamp in quartz is filtered through a soln. of $CoSO_4$ and $NiSO_4$, and gaseous chlorine, all in quartz cells. This source has been used for the detection of the components of nucleic acids.
J. ZÝKA

1607. **Sensitive probe for the Photovolt densitometer.** G. Kronmueller (Dept. of National Health and Welfare, Ottawa, Canada). *J. Chromatography*, 1959, **2** (4), 429–431 (in English).—The sensitivity of a densitometer used for measuring the visible fluorescence, emitted under u.v. light, of spots of polycyclic hydrocarbons on a chromatogram was increased 100-fold by replacing the u.v.-sensitive photocell by a single-crystal CdS cell. Microgram and fractional μg amounts of pyrene give an adequate response.
G. BURGER

1608. **Improved technique for detecting spots on paper chromatograms with iodine vapour.** S. C. Pan (Squibb Inst. for Med. Res., New Brunswick, N.J., U.S.A.). *J. Chromatography*, 1959, **2** (4), 433 (in English).—When iodine vapour is used to stain chromatographic spots, staining of the background can be prevented by first spraying the paper with an aq. soln. of $Al_2(SO_4)_3$. The chromatogram is then dried, exposed to iodine vapour and sprayed with starch soln.
G. BURGER

1609. **Identifying constituents of liquid mixtures.** Benzole Producers, Ltd. [Inventor: A. J. Groszek]. Brit. Pat. 822,432; date appl. 10.12.57.—The difference in the heats of adsorption of the constituents of a fractionated liquid mixture, obtained for example by chromatographic separation, is used to identify the constituents by passing each fraction through an adsorbent (*e.g.*, ion-exchange resin, silica gel, Al_2O_3) and measuring the rise in temp. produced by the adsorption of each fraction.
J. M. JACOBS

1610. **Gas-chromatographic analyser and recorder.** M. Vélut and J. Jourda. *Rev. Inst. Franç. Pétrole*, 1958, **13** (12), 1635–1647.—A detailed account is given of the conversion to automatic operation of a Janak-type apparatus. The eluent gas causes an increased pressure which is transmitted to a micromanometer, movement of which actuates a relay which operates a compensating reservoir, the movement of which is transmitted to a recorder. The apparatus can be used for the rapid analysis of hydrocarbon mixtures up to C_4. N. E.

1611. **Constant-flow device for temperature-programmed gas chromatography.** J. H. Knox (Chem. Dept., Edinburgh Univ., Scotland). *Chem. & Ind.*, 1959, (35), 1085.—Two gas chambers over mercury in a U-tube are connected by a leak tube in which there is a constriction. Pressure from a constant-pressure gas source, 10 to 20 cm of mercury above the max. inlet pressure required by the column, is applied to one chamber and causes a float on the mercury of the other chamber to rise and block the capillary exit from this chamber. The leak ensures the fall of the float, and at equilibrium the rate of flow of the device is that of the leak.
E. G. CUMMINS

1612. **Gas - liquid partition chromatography for metal separations.** H. Freiser (Univ. of Arizona, Tuscon, U.S.A.). *Anal. Chem.*, 1959, **31** (8), 1440.—Both $SnCl_4$ and $TiCl_4$ give well-defined, symmetrical bands when passed through a 5-ft. coiled copper tube, 0·25 in. outside diam., packed with 31% w/w of *n*-hexadecane on Chromosorb. With He as the carrier gas and at a temp. of 102° the retention vol. were 382 ml for $SnCl_4$ and 888 ml for $TiCl_4$.
K. A. PROCTOR

1613. **Reduction of tailing in gas - liquid chromatography.** O. Harva, P. Kivalo and A. Keltakallio (Inst. of Technol., Helsinki, Finland). *Suomen Kem.*, *B*, 1959, **32** (5–6), 71–72 (in English). —Unsymmetrical peaks are obtained when using polar solutes with non-polar stationary liquids (*e.g.*, liquid paraffin) on ground firebrick, Chromosorb or Celite supports. The peaks have been restored to symmetry by the addition to the stationary liquid of 0·1 to 0·5% (w/w) of a surface-active agent, *e.g.*, Span 20, Manoxol OT, stearic acid or glycerol monostearate, in experiments with saturated alcohols, with Celite supports. The partition coefficients were not altered by the additions.
E. G. CUMMINS

1614. **Comparison of detectors for gas chromatography.** I. G. McWilliam (I.C.I. Ltd., Dyestuffs Div., Blackley, Manchester, England). *J. Appl. Chem.*, 1959, **9** (7), 379–388.—The influence of detector sensitivity, response time, and ease of construction and operation on the selection and efficiency of gas-chromatography detectors is discussed. It is advocated that sensitivity should be expressed in terms of the minimum detectable gas concn. (μg per ml) rather than sample size; the base-line noise (μV) and drift (μV per hr.) should also be stated. The response time should be such as to ensure that negligible distortion of the true peak shape occurs. With a symmetrical peak the ratio of the (time) base-width of the peak to the time constant of the detector, and of the peak vol. to the detector vol., should be $\simeq 30:1$. For accurate qual. and quant. analysis, a reproducible and linear detector-output (related to some molecular parameter) is essential. Factors influencing the attainment of this are considered. W. J. BAKER

1615. **Biological objects as detectors in gas chromatography.** E. Bayer and F. Anders (Inst. f. organ. Chem., Tech. Hochsch., Karlsruhe). *Naturwissenschaften*, 1959, **46** (11), 380 (in German).—The active components of the sexual stimulating substance of the female silk moth (*Bombyx mori* L.) were separated by gas chromatography at 91° to 131° by using a mixed stationary phase of silicone high-vacuum grease and Na hexanoate (9:1) with air as carrier gas. The presence of active substances in the gas stream leaving the column was detected by observing the reactions of male silk moths confined in small containers through which the carrier gas passed. Three substances were detected with retention times of 9, 40 and 120 min. The sensitivity was 10^{-10} μg per ml. W. T. CARTER

1616. **Direct spectroscopic examination of electrophoretic zones in agar gel.** G. H. Beaven and W. B. Gratzer (Med. Res. Council Lab., Hampstead, London). *Nature*, 1959, **184** (Suppl. No. 6), 359–360.—Two methods are used. In the first, the zone is transferred to a strip of quartz which is mounted in front of the slit of a logarithmic cam spectrograph. In the second method, which has not been described previously, the agar mounted on quartz is fitted into the cell carriage of a Unicam spectrophotometer, and a strip of plain agar mounted on quartz is used as "blank". The max. difference in extinction between two strips prepared from the same gel was 0·030 at 250 mμ; 12 pairs of strips were examined. The transmission of the agar is increased if the gel is allowed to dry in the cold before spectroscopic examination. A. R. ROGERS

1617. **Improved cutting device for starch-block electrophoresis.** H. Bloemendal and L. Bosch (Dept. of Biochem., Netherlands Cancer Inst., Amsterdam). *Anal. Chem.*, 1959, **31** (8), 1446.—By means of the device described, the cutting is performed in a single manipulation and exactly equal starch segments are obtained, which are easily transferred to centrifuge tubes.
K. A. PROCTOR

See also Abstracts—**1249**, Absorption and pptn. chromatography, literature review. **1429**, High-temp. gas chromatography. **1512**, Plasma proteins on cellulose acetate strips.

Optical

1618. **A burner for cyanogen flame spectroscopy.** Keiichiro Fuwa, R. E. Thiers and B. L. Vallee (Biophys. Res. Lab., Harvard Med. Sch., Mass., U.S.A.). *Anal. Chem.*, 1959, **31** (8), 1419–1421.—The burner described is simple, sturdy, easy to operate and free from hazard. It provides the stability needed under the required conditions of sample flow-rates, and allows accurately controlled

introduction of μl amounts of sample soln. per minute. The flame has a higher excitation energy than other sources, and because of its high sensitivity and analytical precision is very suitable for quant. trace analysis. K. A. PROCTOR

1619. **Electrical modifications of the Baird flame photometer which simplify calibration and improve the accuracy of low potassium readings.** R. C. MacDuffee (William Pepper Lab. of Clin. Med., Univ. of Pennsylvania, Philadelphia, U.S.A.). *Clin. Chem.*, 1959, **5** (5), 492–495.—Simple circuit modifications are described. Serum Na and K can be determined on the same sample dilution with no loss of precision. H. F. W. KIRKPATRICK

1620. **Low-pressure mercury arc for the investigation of light scattering.** M. L. Sosinskil. *Optics and Spectroscopy*, 1959, **6** (3), 254–257; English translation of *Optika i Spektroskopiya*, 1959, **6** (3), 405.—Three types of mercury discharge lamp are described. These have been designed (*i*) to minimise the emission of continuous radiation (*ii*) to produce spectral lines as narrow as possible and (*iii*) to reduce the intensities of the **4348-A** and **4339-A** lines relative to the powerful **4358-A** line. The lamps are suitable as sources for analysis by Raman spectroscopy. B. S. COOPER

1621. **Diffuse reflectance spectrophotometry in the ultra-violet using powdered salts.** T. R. Griffiths, K. A. K. Lott and M. C. R. Symons (Dept. of Chem., Univ. Southampton, England). *Anal. Chem.*, 1959, **31** (8), 1338–1341.—The u.v. spectra of a variety of powders have been studied in the 220 to 400-mμ region. With appropriate precautions, these spectra are trustworthy at least down to 220 mμ. Surface effects are of considerable importance in interpreting the spectra. Certain periodates, iodides, iodates, sodium dithionite, metabisulphite and dithionate, sodium peroxide and ozonide, and chromate and dichromate have been studied to illustrate the potentialities of the technique. The method is simple, and reproducible results are obtained. K. A. PROCTOR

1622. **Double-beam infra-red grating spectrometer.** J. L. Hales (National Chem. Lab., D.S.I.R., Teddington, Middx., England). *J. Sci. Instrum.*, 1959, **36** (6), 264–266.—The compact instrument described has one foreprism, one wavelength cam and two gratings, with 2400 lines per in. in the first order, to cover a wavelength range from 2 to 17 μ. Non-precision components are used without affecting the high degree of resolution. A ratio-recording double-beam system is employed. P. T. BEALE

1623. **The threshold of sensitivity of an infra-red absorption gas analyser with gas modulation.** A. O. Sall' (State Union Construction Bur. of Anal. Instrum.). *Optics and Spectroscopy*, 1959, **6** (3), 247–249; English translation of *Optika i Spektroskopiya*, 1959, **6** (3), 394.—A gas analyser with a single optical path and gas modulation can have a threshold sensitivity very much lower than the double-path type of gas analyser without gas modulation. Gas modulation is achieved either by a periodical change in the length of the sample chamber or by a periodical variation in the gas pressure or by alternately admitting the sample and a standard to the gas chamber at a definite frequency. The radiation detector is tuned to the frequency of modulation. The dependence of the threshold sensitivity of such an analyser on the threshold sensitivity of the detector, on the length of the gas chamber and on the absorption coefficient of the component to be determined has been investigated. B. S. COOPER

1624. **Simple method of producing potassium bromide plates for the infra-red spectroscopy of solids.** A. I. Finkel'shtein, F. L. Malachevskaya, A. M. Fisher and B. G. Rabovskil (Dzorahinski Branch of the Sci. Res. and Planning Inst. of the Nitrogen Ind.). *Optics and Spectroscopy*, 1959, **6** (3), 260–261; English translation of *Optika i Spektroskopiya*, 1959, **6** (3), 415.—The KBr (or NaCl) to be used must be free from KCl. After calcining at 200° for 12 to 18 hr., with grinding every 4 hr., the salt may be stored for an unlimited time in a desiccator over $CaCl_2$. For normal investigations a series of plates were prepared containing 0·1, 0·33, 1 and 3%, respectively, of the material to be studied. If required, an internal standard can also be incorporated in the mixture before pressing. For plates 1 cm square, the compression time is several seconds at a pressure of 10 to 15 tons. B. S. COOPER

1625. **Polyethylene absorption cells for infra-red spectrophotometry.** T. Robinson (Biological and Food Research Center, Syracuse Univ., Syracuse, N.Y.). *Nature*, 1959, **184** (Suppl. No. 7), 448–449.—Make a bag of polyethylene by heat-sealing, around three sides, two sheets each 0·002 in. thick. Add a few drops of the liquid sample and squeeze out the excess to remove air bubbles and leave a capillary film. Polyethylene shows strong absorption at 3·4 μ, 6·8 μ and 13·5 to 14 μ, but is otherwise almost transparent. The cells may be used for qual. examination of aq. soln. A. R. ROGERS

Thermal

1626. **Melting-point apparatus for simultaneous observation of samples in transmitted and reflected light.** H. E. Ungnade, E. A. Igel and B. B. Brixner (Univ. of California, Los Alamos Sci. Lab., N. Mex., U.S.A.). *Anal. Chem.*, 1959, **31** (8), 1432–1434.—In the apparatus described, use is made of an electrically heated copper block and an optical system which projects both transmitted and reflected images of the sample side by side on a rear projection screen. It is particularly suitable for use with explosive materials. K. A. PROCTOR

1627. **Use of thermistors in the ebullioscopic determination of molecular weights.** J. G. van Pelt (N. V. Philips-Duphar, Weesp, Netherlands). *Philips Tech. Rev.*, 1958–1959, **20** (12), 357–361.—The high sensitivity and low thermal inertia of thermistors have been applied in the double-cell ebullioscopic method (described) to determine molecular weights of $\simeq$500 with an accuracy better than 1%. Details of the modification of standard thermistors and the circuit diagrams are given. The error in the temp. measurement is $\ngtr$0·0003°. E. G. CUMMINS

1628. **Simple apparatus for the micro-determination of methane.** S. Toby (Rutgers Univ., New Brunswick, N.J., U.S.A.). *Anal. Chem.*, 1959, **31** (8), 1444–1445.—A simple apparatus is described

with which the complete combustion of a 30-μmole sample is achieved at a temp. of 670° within 2 hr. The error is $< \pm 0{\cdot}3\%$. G. P. Cook

1629. **Thermometric titration in fused salts.** J. Jordan, J. Meier, E. J. Billingham, jun., and J. Pendergrast (Dept. of Chem., Pennsylvania State Univ., University Park, U.S.A.). *Anal. Chem.*, 1959, **31** (8), 1439–1440.—A method has been developed for performing automatic thermometric titrations, involving pptn., complexing, and oxidation - reduction processes, in molten salts. The instrumentation is basically similar to that required for conventional aqueous titrimetry and is adapted to fused salt media by using an automatic vertical syringe micro-burette, completely enclosed in the top portion of an argon-filled gas space in an adiabatic titration cell. K. A. Proctor

1630. **Infra-red heating applied to differential thermal analysis.** J. A. Hill and C. B. Murphy (Gen. Engng Lab., General Electric Co., Schenectady, N.Y., U.S.A.). *Anal. Chem.*, 1959, **31** (8), 1443–1444.—The heating system described is simple and inexpensive to construct and is satisfactory for obtaining differential thermal curves of organic compounds when data below 240° are required. The equipment is portable. K. A. Proctor

Electrical

1631. **Determination of [electrical] conductivities using unconventional electrode systems.** V. S. Griffiths (Chem. Dept., Battersea Coll. of Technol., London, England). *Talanta*, 1959, **2** (3), 230–234.—An apparatus for measuring electrolytic conductivity with non-contacting electrodes and a three-terminal transformer ratio-arm bridge is described and illustrated. With a capacity cell (illustrated) and the bridge used as a "dielectric bridge," determinations can be made of impurities, *e.g.*, H_2O, in organic liquids such as acetone. The accuracy is satisfactory up to 5% of H_2O. W. J. Baker

1632. **Simple and inexpensive polarograph cell.** J. L. Monkman (Dept. of National Health and Welfare, Ottawa, Canada). *Anal. Chem.*, 1959, **31** (8), 1445.—The borosilicate-glass cells described are rugged, easily cleaned, and simple and inexpensive to make. The total capacity is $\simeq$ 5 ml. K. A. Proctor

1633. **Electrode holder for work in controlled atmospheres.** A. M. Pommer and J. F. Abell (U.S. Geolog. Survey, Washington, D.C., U.S.A.). *Anal. Chem.*, 1959, **31** (8), 1443.—Methyl methacrylate plastic closures are described which are suitable for holding electrodes or other devices in vessels in which pH or other measurements are being carried out in a controlled atmosphere. K. A. Proctor

1634. **Use of an aluminium electrode in potentiometric titrations.** I. Pražák and J. Grimmer (Paint and Varnish Res. Institute, Prague). *Coll. Czech. Chem. Commun.*, 1959, **24** (6), 2049–2051 (in German).—An aluminium indicator electrode is suitable for potentiometric neutralisation titrations in aqueous and non-aqueous media [99% ethanol, or benzene - ethanol (1:1)] and its potential changes are greater than those of the antimony electrode. The nature of the electrode surface is the decisive factor. The electrode was prepared from a sheet of pure aluminium, 2 mm thick, and was 20 mm long and 2 mm wide, with a slender shaft 100 mm long. The electrode was polished mechanically and then washed in boiling pure CCl_4. Strong and weak acids can be successfully titrated. Good results were obtained when titrating benzoic and oleic acids in non-aqueous media. J. Zýka

1635. **Voltammetric, potentiometric and amperometric studies with a rotated aluminium-wire electrode. I. Voltammetric behaviour of the R. Al. E.** I. M. Kolthoff and C. J. Sambucetti (Sch. of Chem., Univ. of Minnesota, Minneapolis, U.S.A.). *Anal. Chim. Acta*, 1959, **29** (1), 17–24 (in English).—A rotated aluminium-wire electrode is described. The electrode is readily polarisable both cathodically and anodically, and is depolarised by OH^- and F^-. The potential of the electrode is very sensitive to the concn. of F^-. The electrode functions as an OH^- indicator electrode at a pH >9, and as an F^- indicator electrode in acid medium. T. R. Andrew

1636. **Chloride-free reference electrode for use in acetic acid solvent.** N. Al-Qaraghuli and K. G. Stone (Chem. Dept., Michigan State Univ., E. Lansing, U.S.A.). *Anal. Chem.*, 1959, **31** (8), 1448.—The electrode described is based on the mercury - mercurous acetate couple in acetic acid, and reproducible e.m.f. values *vs.* a platinum measuring electrode were observed for the Ce^{IV} - Ce^{III}, Fe^{III} - Fe^{II}, Ti^{IV} - Ti^{III}, and Cr^{VI} - Cr^{III} systems. All the titrations were carried out in the presence of $HClO_4$ and no effects due to changes in acid concn. during the titration were noted. None of the difficulties experienced with calomel electrodes was observed. K. A. Proctor

Errata

February (1960) issue, abstract 458, line 4. *For* Fujimaga *read* Fujinaga.

February (1960) issue, abstract 575, 5th line from end. *For* citreonellal *read* citronellal.

February (1960) issue, abstract 586, line 8. *For* K - phthalate *read* K H phthalate.

February (1960) issue, cross references at end of Section 3, line 7. *For* **896** *read* **806**.

February (1960) issue, abstract 672, line 9. *For* ethanolic-fluoro-2:4-dinitrobenzene *read* ethanolic 1-fluoro-2:4-dinitrobenzene.

ABBREVIATIONS

Certain abbreviations in everyday use are not included in the following list. When any doubt might arise from the use of an abbreviation or symbol the word is printed in full.

alternating current	a.c.
ampere	amp.
Ångström unit	Å
anhydrous	anhyd.
approximate, -ly	approx.
aqueous	aq.
atmospher-e, -ic	atm.
boiling-point	b.p.
British thermal unit	B.Th.U.
calorie (large)	kg-cal.
calorie (small)	g-cal.
centimetre	cm
coefficient	coeff.
Colour Index	C.I.
concentrated	conc.
concentration	concn.
constant	const.
crystalline, crystallised	cryst.
cubic	cu.
current density	c.d.
cycles per second	c/s
density	ρ
density, relative	d or wt. per ml
dilute	dil.
direct current	d.c.
distilled	dist.
ethylenediaminetetra-acetic acid	EDTA
electromotive force	e.m.f.
equivalent	equiv.
gram	g
gram-molecule	mole
half-wave potential	$E_{\frac{1}{2}}$
hour	hr.
hydrogen ion exponent	pH
infra-red	i.r.
insoluble	insol.
international unit	i.u.
kilogram	kg
kilovolt	kV
kilowatt	kW
liquid	liq.
maxim-um, -a	max.
melting-point	m.p.
microgram	μg (not γ)
microlitre	μl
micromole	μmole
micron	μ
milliampere	mA
milli-equivalent	milli-equiv.
milligram	mg
millilitre	ml
millimetre	mm
millimicrogram	mμg
millimolar	mM
millivolt	mV
minute (time)	min.
molar (concentration)	M
molecul-e, -ar	mol.
normal (concentration)	N
optical rotation	α_λ^t
ounce	oz
parts per million	p.p.m.
per cent.	%
per cent. (vol. in vol.)	% (v/v)
per cent. (wt. in vol.)	% (w/v)
per cent. (wt. in wt.)	% (w/w)
potential difference	p.d.
precipitate (as a noun)	ppt.
precipitated	pptd.
precipitating	pptg.
precipitation	pptn.
preparation	prep.
qualitative, -ly	qual.
quantitative, -ly	quant.
recrystallised	recryst.
refractive index	n_λ^t
relative band speed	R_F
relative humidity	r.h.
revolutions per minute	r.p.m.
saponification value	sap. val.
saturated calomel electrode	S.C.E.
second (time)	sec.
soluble	sol.
solution	soln.
specific gravity	sp. gr.
specific rotation	$[\alpha]_\lambda^t$
square centimetre	sq. cm.
standard temp. and pressure	s.t.p.
temperature	temp.
ultra-violet	u.v.
vapour density	v.d.
vapour pressure	v.p.
volt	V
volume	vol.
watt	W
wavelength	λ
weight	wt.

In addition, the following symbols may be used in conjunction with numerical values or in mathematical expressions—

greater than	$>$
not greater than	$\ngtr$
is proportional to	$\propto$
less than	$<$
not less than	$\nless$
of the order of, approximately	$\simeq$

The principal Pharmacopoeias are denoted by B.P., U.S.P. or D.A.B., together with the identifying roman numeral or year.

Valency states are represented by a superscript roman numeral, *e.g.*, Fe^{II}, Mo^{V}. Substances in the ionic state are represented by Na^+, Fe^{2+}, Fe^{3+}, etc., for cations and by Cl^-, SO_4^{2-}, PO_4^{3-}, etc., for anions.

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